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FINAL REPORT

PROPERTIES OF POWDERED TITANIUM ALLOYS

by

Gerald Friedman

WHITTAKER CORPORATION
Nuclear Metals Division
West Concord, Massachusetts

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

15 May 1969

Contract NAS3-10301

NASA Lewis Research Center
Cleveland, Ohio

John Kazaroff, Project Manager

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Scientific and Technical Information Facility
P. O. Box 333
College Park, Maryland 20740

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ABSTRACT

Titanium alloy powders Ti-6Al-4V and Ti-5Al-2.4Sn made by five different techniques were compared with respect to chemistry and physical characteristics. The powders were hot pressed over a range of temperatures and pressures, and the solid bodies thus formed were further analyzed and compared. The Rotating Electrode powder-making process was selected as the most suitable technique for producing the required low-oxygen powders, and additional quantities of powder were prepared by this method. Large blocks were hot-pressed to full density from the two alloy powders. Samples taken from the blocks were tested in tension and under plain-strain conditions at room and at cryogenic temperatures, in the longitudinal and the transverse directions. The powder specimens were compared to wrought samples tested under the same conditions and showed tensile properties equal to or better than their wrought counterparts at all temperatures. No comparison could be made between the wrought and powder fracture toughness specimens, since all but one series of tests produced invalid test results. Recommendations for additional work to provide valid fracture toughness conditions are included.

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PROPERTIES OF POWDERED TITANIUM ALLOYS

By Gerald Friedman
Whittaker Corporation, Nuclear Metals Division

SUMMARY

Low oxygen (ELI) Ti-6Al-4V and Ti-5Al-2.5 Sn forged bar was converted to powder by the hydride-dehydride process, mechanical attritioning of chips, the Rotating Electrode Process, and by a chemical coreduction procedure. Evaluation of the powders showed that only the spherical powders made by the Rotating Electrode technique retained oxygen contents below 1000 ppm. The irregular powders made by the other techniques had oxygen contents ranging from 1220 (attrited chips) to 8100 ppm (coreduced). The powders were compacted at 1450, 1650, and 1850°F, 50, 75, and 100 tsi. Except for the coreduced powders, all samples were over 99 percent dense at a pressure/temperature combination of 75 tsi and 1650°F, or higher. Analysis of the various compacts showed that only those made from the Rotating Electrode Process powder had not oxidized further. Excluding the coreduced powder compacts, the oxygen contents in the compacts made by the remaining techniques now ranged from 1570 ppm (hydride-dehydride) to 2800 ppm for one of the attrited powders.

Tensile test results coupled with the oxygen analyses led to the selection of the Rotating Electrode Process powder compacts for further study. Heat treated Ti-6Al-4V samples had these properties: 132-140,000 psi UTS, 124-131,000 YS, 11-10 percent elongation, and 44 - 32 percent RA. For the Ti-5Al-2.5Sn alloy, the comparable values were 122-128,000 psi UTS, 113-118,000 psi YS, 11-19 percent elongation, and 26-45 percent RA.

Additional sections of the forged bars used in the first phase of the program were extruded to electrode stock and converted to powder by the Rotating Electrode Process. The two powder alloys were then hot compacted into 6-inch by 6-inch by 3-inch blocks at 1850°F and 70 tsi. The blocks were annealed and then sectioned into longitudinal and transverse tensile and plain-strain fracture toughness specimens. A similar number of samples was also prepared from the as-received forged bar.

Tensile tests were run on triplicate flat samples in longitudinal and transverse directions, at room temperature, -320°F, and -423°F. The Ti-6Al-4V powder alloy had mechanical properties in both longitudinal and transverse direction that were equal to or better than those for the wrought bar. This was also true for the Ti-5Al-2.5Sn alloy, in the transverse direction, at all temperatures. In the longitudinal direction the powder alloy properties were similar to or slightly inferior to those of the wrought alloy.

Plain-strain fracture toughness tests were performed on notched pre-cracked flat specimens, following the ASTM recommended practice. Only the powder Ti-6Al-4V specimens tested at -423°F produced valid results. K_{IC} values for these specimens averaged 69,700 psi $\sqrt{in}$ in the longitudinal direction and 75,600 psi $\sqrt{in}$ in the transverse direction. The tests of the wrought counterparts of these specimens were not valid, and so no comparison of fracture toughness properties between the wrought and powder specimens is possible.

INTRODUCTION

This report describes a program performed to evaluate the strength and toughness of hot pressed titanium alloy powders at room and at cryogenic temperatures. The program objective was to determine how the mechanical properties of solid bodies formed from powder would compare with wrought specimens of the same size and with the same chemical analysis. Such information would prove valuable in determining the usefulness of powder techniques for the fabrication of parts that are difficult or costly to make by conventional techniques. The basis for this expectation was the belief that the powder process would produce fine-grained structures possessing a high degree of chemical homogeneity and property uniformity.

In the first part of the program titanium alloy powders made by five different techniques were compared. Following an evaluation based on powder and solid characteristics, one powder type was selected for use in the balance of the program. Rectangular blocks, 6 inches by 6 inches by 3 inches, of both alloys were pressed, and samples cut from the blocks were tested at room temperature, -320°F, and -423°F.

TECHNICAL PROGRAM

Sample Preparation and Testing

Ti-6Al-4V and Ti-5Al-2.5Sn ELI grade (Extra-Low Interstitial Content) forged bars were purchased by Nuclear Metals. Sections of each bar were sent to Nuclear Materials and Equipment Corporation, Apollo, Pennsylvania, for conversion to powder by both the hydride-dehydride process (Numec Hyd) and by mechanical attrition (Numec MA). In the former process, powder is prepared by enclosing the titanium bar in a sealed chamber which is then evacuated and back-filled with hydrogen as the chamber temperature is raised. Whereas titanium is capable of dissolving nearly 8 atomic percent of hydrogen at 600°F, the reacted mass of metal can retain only from 0.05 to 0.14 atomic percent hydrogen at temperatures below 250°F. The excess hydrogen is therefore forced out of solution at these lower temperatures and is present as a titanium hydride phase. At levels above 200 ppm, the precipitated hydrogen embrittles the matrix, thus making possible a relatively simple crushing operation. The powder which results from crushing the titanium hydride is then converted back to the metallic state by a subsequent vacuum heat treatment.

In the mechanical attrition process, the forged bar was machined to chips, which were then converted to powder in a hammer mill. The machining and milling operations were performed in a helium atmosphere.

Dominion Magnesium, Ltd., Toronto, Canada (Dom), prepared ten pounds of Ti-6Al-4V powder by the coreduction of titanium, aluminum, and vanadium compounds.

Ten pounds of Ti-5Al-2.5Sn powder were procured from the Penn-Nuclear Corporation, Penn, Pennsylvania (Penn) which used a combination gas impingement and mechanical attrition technique to produce powder from their own starting stock.

Ten pounds of each alloy powder were prepared at Nuclear Metals by their Rotating Electrode Process (REP). Portions of the same forged bars sent to Numec were extruded, cut into 10-inch lengths, and finish machined to 1-1/4-inch diameter rods. The metal rods were loaded into an 8-foot diameter tank which was subsequently evacuated and back-filled with helium. By means of a glove-port in the tank wall, each bar was positioned in turn in the chuck of a high-speed spindle inside the tank. When the electrode had attained the desired rotational speed, an arc was struck between the face of the titanium bar and a non-rotating tungsten electrode. The combined action of the arc and the rotating electrode results in the formation of spherical droplets which fly off from the electrode face. The droplets freeze in flight into spherical powder particles and are completely solid before they drop to the floor of the tank.

Powder evaluation began with the transfer of each of the eight powders in turn to the inert-gas glove box. Each ten-pound powder lot was then passed through a sample splitter so that representative samples could be taken for the various tests. The samples and the balance of the powder from each vendor were then removed from the glove box in sealed containers. Shadowgraphs of the various powder types are shown in Figure 1.

Sealed vials of powder were sent to an independent analytical laboratory* for chemical analysis. Comparing the results (Table I) of the chemical analysis with the appropriate specification,** we see that: (1) the Dominion Ti-6Al-4V powder is grossly out of specification with respect to oxygen (8100 ppm), carbon (940 ppm), hydrogen (2960 ppm), and vanadium (2.7 percent). The high interstitial content of this powder explains its poor performance in the hot pressing tests (see below); (2) only the Nuclear Metals Rotating Electrode powders meet ELI specification for titanium. The Numec powders were made from the same low-oxygen forged bars that Nuclear Metals used, but the oxygen level in Numec's four powders ranges from 1220 to 1560 ppm.

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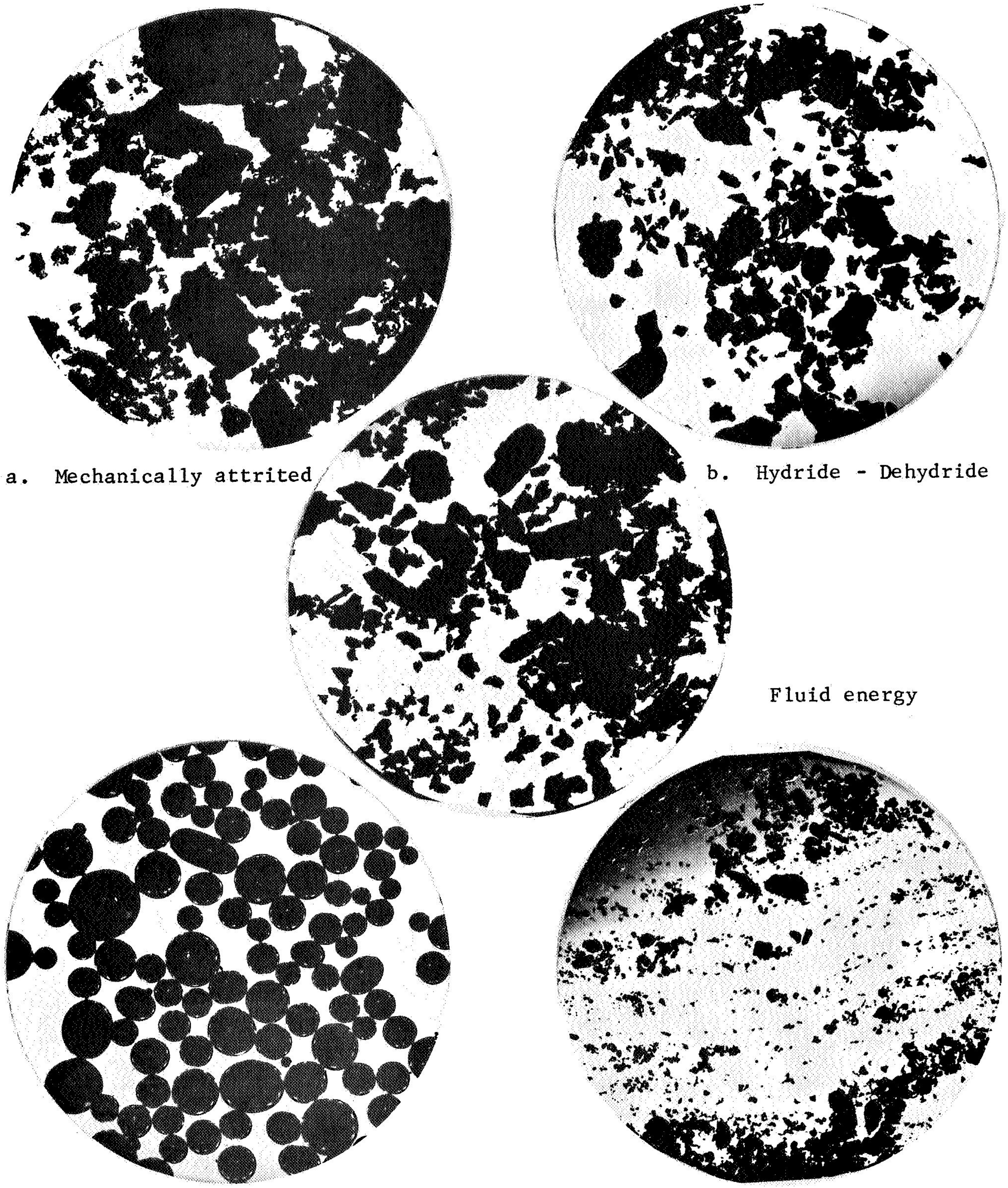
*Ledoux and Company, Teaneck, New Jersey.

**MIL-T-9047D, "Titanium Alloy Bars, Forgings, and Forging Stock," 9 June 1967.



a. Mechanically attrited

b. Hydride - Dehydride

Fluid energy

d. Rotating electrode

e. Coreduction

Figure 1. Titanium alloy powders. 25X

TABLE I.— CHEMICAL ANALYSES FOR EIGHT TITANIUM ALLOY POWDERS

A. Ti-6Al-4V	Al (%)	V (%)	Fe (%)	O (ppm)	C (ppm)	N (ppm)	H (ppm)
Sample							
MA[a]	5.84	4.19	0.16	1560	130	189	98
REP[a]	5.67	4.30	0.15	750	67	146	58
Co-reduced	5.72	2.70	0.07	8100	940	187	2960
Hyd[a]	5.94	4.32	0.21	1300	61	155	213
6 inch forged bar	6.31	4.27	0.11	700	270	70	16
MIL Spec., Ti-6Al-4V[b]	5.50-6.75	3.50-4.50	0.30 (max)	2000 (max)	1000 (max)	500 (max)	150 (max)
MIL Spec., Ti-6Al-4V, ELI	5.50-6.75	3.50-4.50	0.25 (max)	1300 (max)	800 (max)	500 (max)	125 (max)

B. Ti-5Al-2.5Sn	Al (%)	Sn (%)	Fe (%)	O (ppm)	C (ppm)	N (ppm)	H (ppm)
Sample							
REP[c]	5.25	2.49	0.20	750	27	106	39
Fluid energy	5.16	2.45	0.20	2800	96	523	187
Hyd[d]	4.93	2.41	0.04	1380	22	149	56
MA[d]	4.98	2.40	0.07	1220	42	191	90
6-1/2 inch forged bar	5.46	2.80	0.02	740	60	140	6
1-1/4 inch ground bar	5.50	2.40	0.25	950	100	100	60
MIL Spec., Ti-5Al-2.5Sn	4.0-6.0	2.0-3.0	0.50 (max)	2000 (max)	1500 (max)	700 (max)	200 (max)
MIL Spec., Ti-5Al-2.5Sn, ELI	4.25-5.75	2.0-3.0	0.25 (max)	1200 (max)	800 (max)	700 (max)	125 (max)

[a]These three powders were made from the 6 inch forged bar.

[b]MIL-T-9047, "Titanium Alloy Bars, Forgings, and Forging Stock", 9 June 1967.

[c]Made from 1-1/4 inch ground bar to replace accidentally contaminated batch of powder made from the 6-1/2 inch bar.

[d]These two powders made from the 6-1/2 inch forged bar.

The fluid-energy milled powder, containing 2800 ppm oxygen, exceeds the limit for standard grade Ti-5Al-2.5Sn.

Based on these data, the powders can be placed in three categories: In the first group are the ELI powders, with very low oxygen contents. In the second group are powders with approximately twice the oxygen in the first group; these powders are still within specification for normal titanium alloys. Powders in the third group have very high oxygen contents.

The REP powders fall into the first category, the hydride-dehydride and mechanically attrited powders into the second, and the fluid-energy milled and coreduced powders constitute the third group.

Additional samples obtained from the sample splitter[1] were used to determine flow rate, apparent density, and particle size distribution. Flow rate and apparent density determinations were carried out in accordance with the appropriate ASTM specifications:

	Flow Rate[2] (seconds/50g)	Apparent Density[3] (g/cc)	(% of theo.)
1. Ti-6Al-4V			
REP	24	2.72	61.5
MA	No flow	1.13	25.6
Hyd	44.5	1.65	37.2
Coreduced	No flow	1.29	29.2
2. Ti-5Al-2.5Sn			
REP	22	2.83	63.5
MA	No flow	1.52	34.2
Hyd	51	1.51	34.0
Fluid-energy	44	1.79	40.2

These two tests illustrate a major difference between the irregular and the spherical powders. The angular-blocky coreduced and hydride and mechanically attrited powders are much more "fluffy" than the REP powder, and in fact occupy twice the volume for equal weights. The blocky-particle powders have a strong tendency to bridge over, which accounts for their poor

1. A sample splitter, or riffler, reduces a large powder sample to a quantity suitable for testing purposes, while still retaining in the small "split" sample the same proportion of fine and coarse powder. The splitter divides the sample into halves, and by repeating the operation the sample can then be split into quarters, eighths, sixteenths, etc., as desired.

2. B 213-48, "Flow Rate of Metal Powders," ASTM.

3. B 212-48, "Apparent Density of Metal Powders," ASTM.

flow characteristics in the Hall Flowmeter. The REP spherical powders flow very easily.

Sieve analyses were determined by slightly modifying the ASTM technique. Instead of obtaining an exact 100 g sample of each powder, the split closest to 100 g was used. In this way, a truly representative sample of powder was obtained from each lot, and the risk of analyzing a non-representative sample was avoided. The sieve analyses and size distribution curves for the four powders are presented in the attached data sheets, Figures 2-5.

Each powder was then cold compacted in an arrangement comprising an inert gas glove box containing a 2-inch I.D. steel packing die, a 1-1/4-inch diameter punch, and an O-ring-sealed piston driven by an electro-hydraulic unit located beneath the box (Figure 6). Ten carbon steel compaction cans were loaded into the box with each lot of powder. The cans were 2-inch O.D. by 1-3/4-inch I.D. by 5 inches long, with an end plate welded across one end. The end plate welds were all leak checked with a helium mass spectrometer before being transferred into the glove box.

The compacts were prepared by filling each can with powder and transferring it to the packing die, where it was pressed for 10 seconds at 1000-1400 psi. More powder was added to the can and pressure was again applied. These steps were repeated until each can contained approximately one pound of powder.

The compacts were then encased in individual rubber bags and were transferred to a welding box where the top lid, with steel evacuation tube attached, was joined to the can body. This weld was also checked for leaks with the helium mass spectrometer. The sound billets were then connected to a vacuum pump and were heated to 800°F to drive off adsorbed gasses. The billets were evacuated overnight and were then sealed off by forge welds on the steel evacuation tubes.

The compacts from each powder vendor were then hot pressed at 1450, 1650, and 1850°F under pressures of 50, 75, and 100 tons per square inch. Each compact was pressed individually, using the arrangement illustrated in Figure 7. The extrusion press container, ram, and the hardened punches were maintained at 900°F, a temperature which minimizes thermal shock to the tooling while maintaining full tool hardness. Transfer time from the billet-heating furnace to the container of the 300 ton press averaged 10-15 seconds; placement of the backer plate and application of ram pressure required approximately 5 additional seconds. Pressure was maintained on the billet for approximately 10 seconds.

The different bulk densities of the various powders combined with their differing cold-pressing characteristics, yielded compacts of differing final lengths for initially identical billets.

Standard Screen Scale

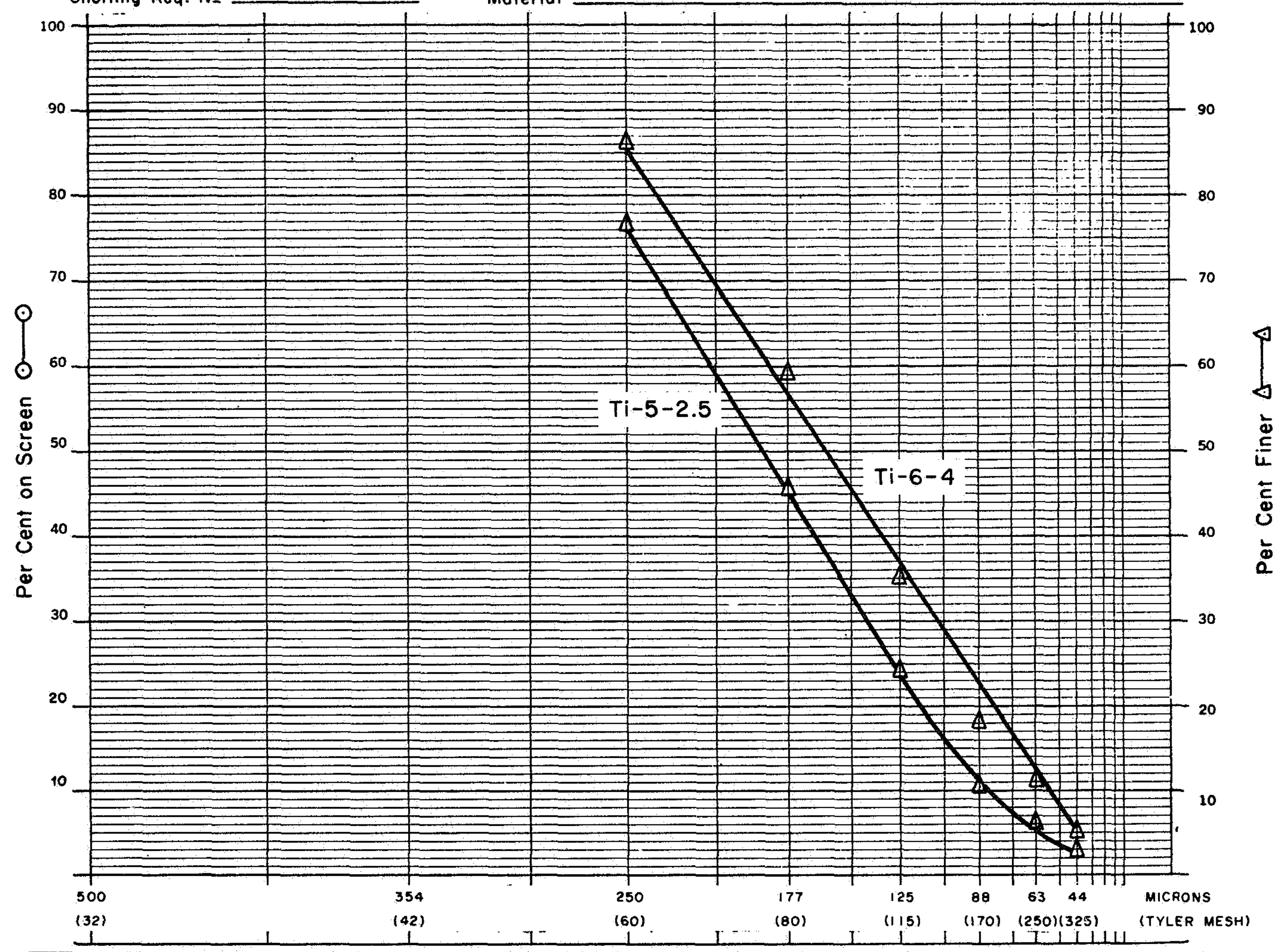
Job Nº 321-0002 Electrode Dia. (in.) —

Date 11-14-67 Speed (rpm) —

Name NUMEC MECHANICAL ATTRITED POWDER

Shotting Req. Nº

Material Ti-6Al-4V & Ti-5Al-2.5Sn



	Printed Screen Scale Ratio 1.414				Ti-6Al-4V			Ti-5Al-2.5Sn		
	Openings		Mesh							
	Microns	Millimeters	Tyler	U.S.	Sample Weights	Per Cent	Per Cent Finer	Sample Weights	Per Cent	Per Cent Finer
	500		32	35						
					INITIAL WT. = 81.50g			INITIAL WT. = 77.15g		
	354		42	45						
ON	250		60	60	11.28	13.84	86.16	18.31	23.61	76.38
ON	177		80	80	22.14	27.17	58.99	23.88	30.79	45.59
ON	125		115	120	19.73	24.20	34.79	16.94	21.85	23.74
ON	88		170	170	14.02	17.20	17.59	10.22	13.18	10.56
ON	63		250	230	5.67	6.95	10.64	3.37	4.34	6.22
ON	44		325	325	4.54	5.57	5.07	2.48	3.19	3.03
ON	PAN				4.13 (1)	5.07	—	2.35 (1)	3.03	—
				Totals,	81.50	100			99.99	

(1) INCLUDES 0.14g LOST (Ti-6Al-4V)

(1) INCLUDES 0.05g LOST (Ti-5Al-2.5Sn)

DATA

Total Shot Weight

Split Sample Yes

No

Sample Weight gms.

Remarks

Figure 2. Sieve analysis for Numec MA Ti-6Al-4V and Ti-5Al-2.5Sn.

Standard Screen Scale

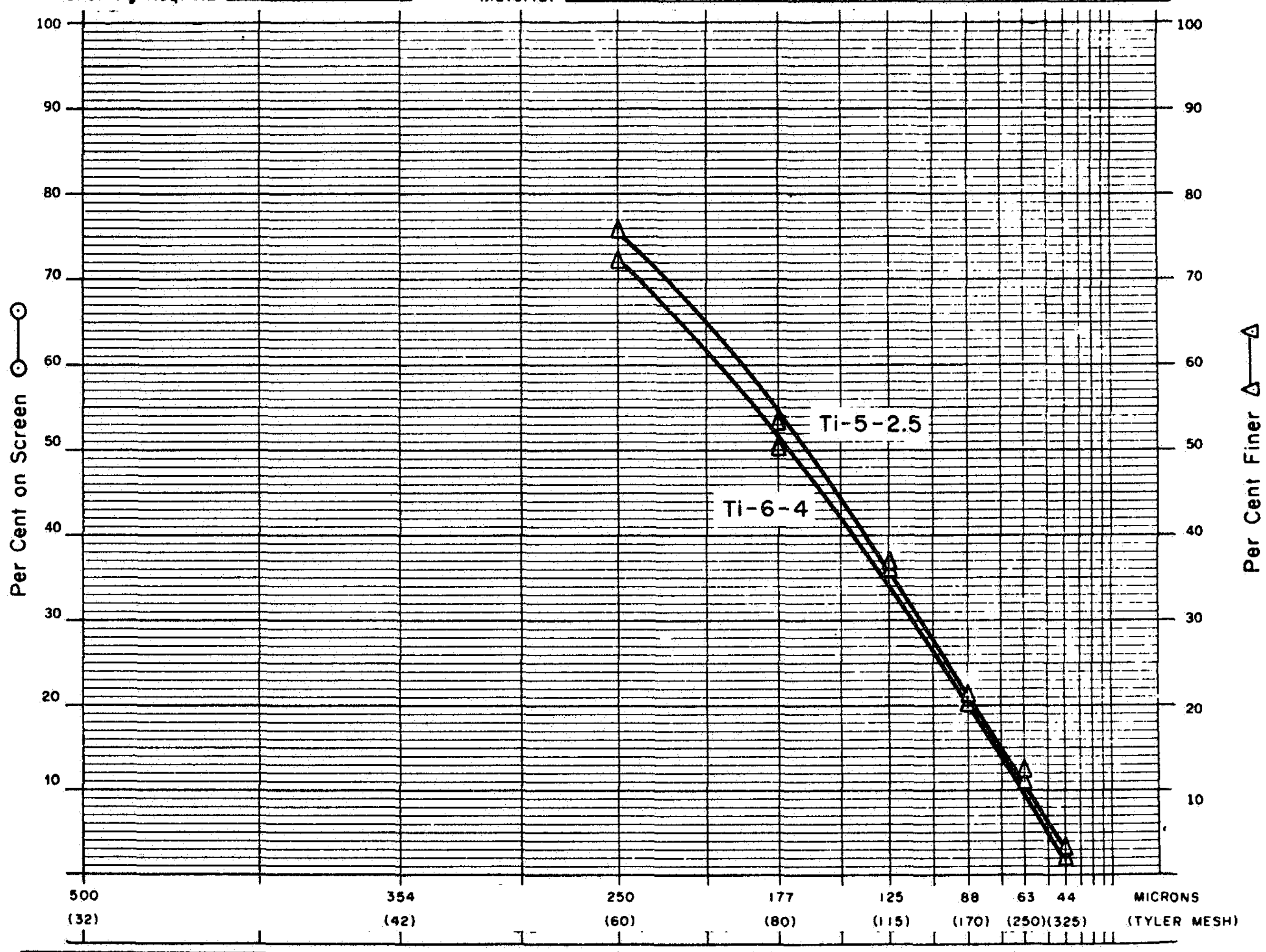
Job No 321-0002 Electrode Dia. (in.) —

Date 12-5-67 Speed (rpm) —

Name NUMEC HYDRIDE-DEHYDRIDE POWDER

Shotting Req. No

Material Ti-6Al-4V & Ti-5Al-2.5Sn



	Printed Screen Scale Ratio 1.414				Ti-6Al-4V			Ti-5Al-2.5Sn		
	Openings		Mesh							
	Microns	Milli-meters	Tyler	U.S.	Sample Weights	Per Cent	Per Cent Finer	Sample Weights	Per Cent	Per Cent Finer
	500		32	35						
					INITIAL WT. = 134.157g			INITIAL WT. = 59.055g		
	354		42	45						
ON	250		60	60	37.60	28.02	71.98	14.50	24.56	75.44
ON	177		80	80	28.98	21.60	50.38	13.02	22.05	53.39
ON	125		115	120	19.26	14.36	36.02	10.40	17.61	35.78
ON	88		170	170	20.97	15.63	20.39	9.00	15.24	20.54
ON	63		250	230	11.32	8.44	11.95	4.84	8.20	12.34
ON	44		325	325	11.92	8.89	3.06	5.26	8.91	3.43
ON	PAN				4.11 (1)	3.06	—	2.03	3.43	—
				Totals,	134.15		99.99	59.05		99.98

DATA

Total Shot Weight

Split Sample Yes

No

Sample Weight gms.

Remarks

Figure 3. Sieve analysis for Numec Hyd Ti-6Al-4V and Ti-5Al-2.5Sn.

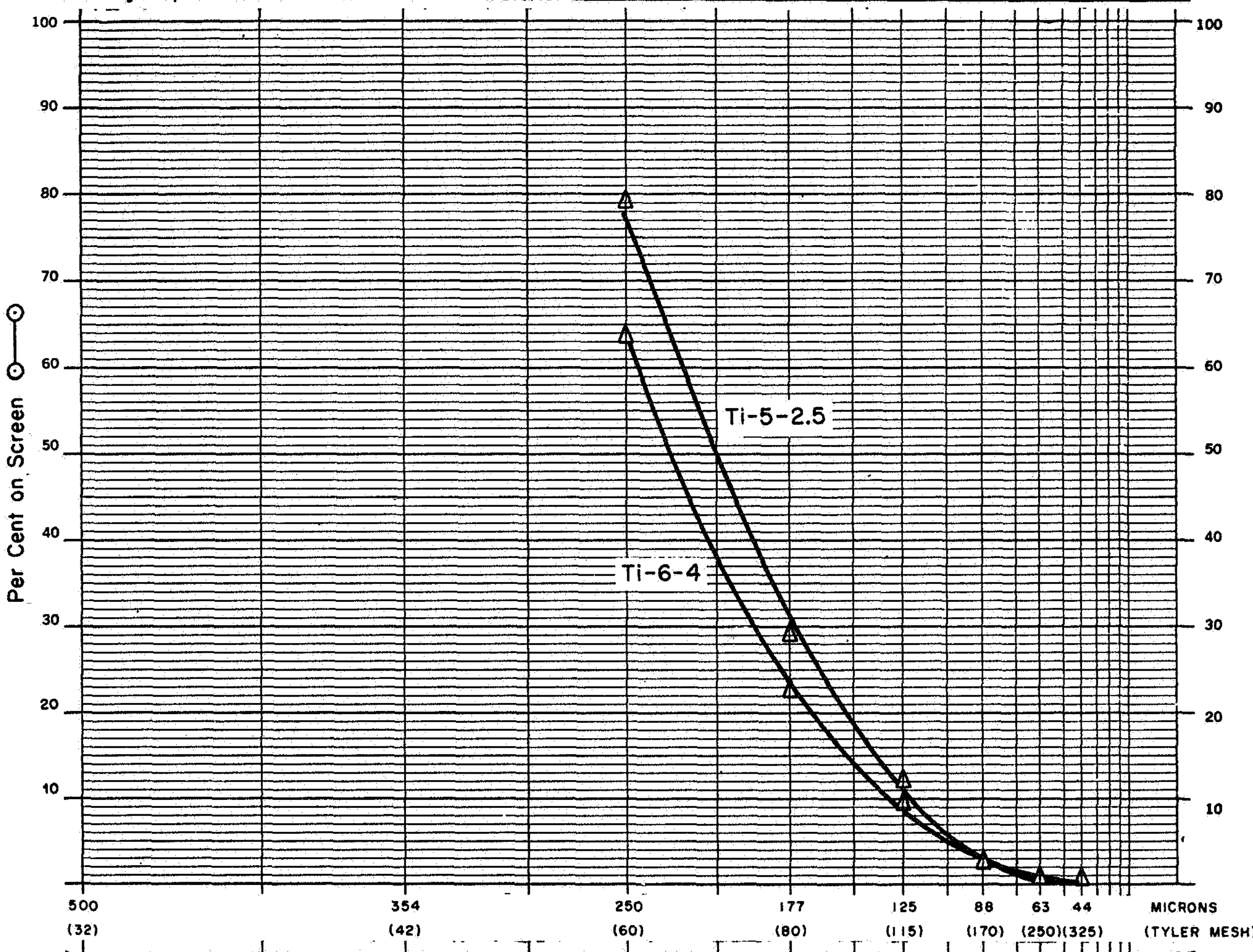
Standard Screen Scale

Job No 321-0002 Electrode Dia. (in.) 1 & 1 1/8" DIA.

Date 11-14-67 Speed (rpm) 15-20,000

Name NUCLEAR METALS ROTATING ELECTRODE POWDER

Shotting Req. No ______ Material Ti-6Al-4V & Ti-5Al-2.5Sn



	Printed Screen Scale Ratio 1.414				Ti-6Al-4V			Ti-5Al-2.5Sn		
	Openings		Mesh							
	Microns	Millimeters	Tyler	U.S.	Sample Weights	Per Cent	Per Cent Finer	Sample Weights	Per Cent	Per Cent Finer
	500		32	35						
					INITIAL WT. = 125.89g			INITIAL WT. = 160.79g		
	354		42	45						
ON	250		60	60	45.65	36.26	63.74	33.77	21.01	78.99
ON	177		80	80	51.52	40.93	22.81	79.70	49.50	29.42
ON	125		115	120	16.56	13.15	9.66	28.09	17.47	11.95
ON	88		170	170	9.27	7.36	2.30	15.01	9.33	2.62
ON	63		250	230	1.91	1.52	0.78	2.80	1.74	0.88
ON	44		325	325	.54(1)	0.43	0.35	0.79	0.49(1)	0.39
ON	PAN				.45	0.35	—	0.63	0.39	—
				Totals,	125.89	100.00		160.79	100	
					(1) INCLUDES LOSS OF 0.36g			(1) INCLUDES LOSS OF 0.42g		

DATA

Total Shot Weight ______

Split Sample Yes ______ No ______

Sample Weight ______ gms.

Remarks

Figure 4. Sieve analysis for Nuclear Metals REP Ti-6Al-4V and Ti-5Al-2.5Sn.

Standard Screen Scale

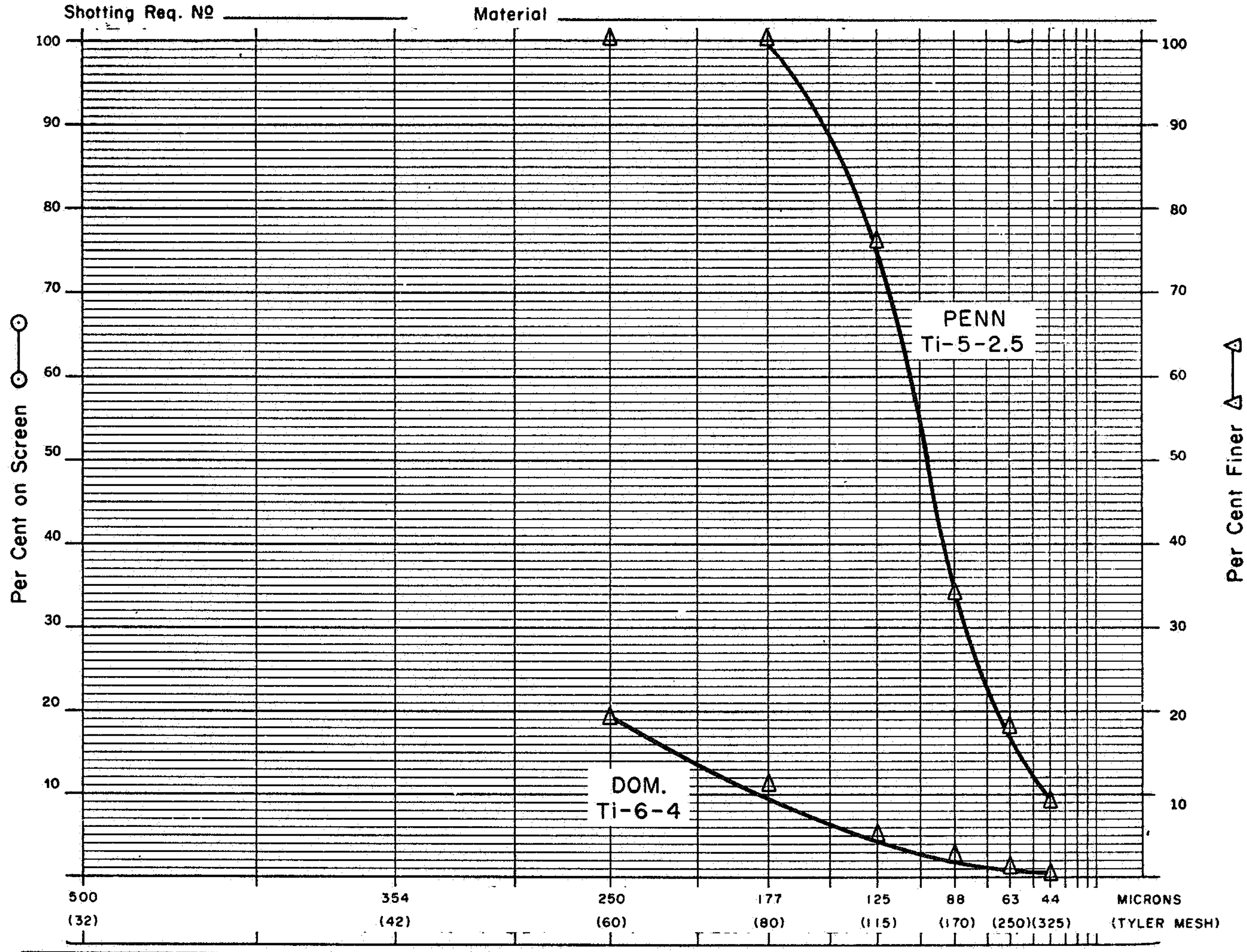
Job № 321-0002 Electrode Dia. (in.) —

Date 12-12-67 Speed (rpm) —

Name DOMINION Ti-6Al-4V & PENN NUCLEAR Ti-5Al-2.5Sn

Shotting Req. №

Material



	Printed Screen Scale Ratio 1.414				DOM. Ti-6Al-4V			PENN Ti-5Al-2.5Sn		
	Openings		Mesh							
	Microns	Millimeters	Tyler	U.S.	Sample Weights	Per Cent	Per Cent Finer	Sample Weights	Per Cent	Per Cent Finer
	500		32	35						
					INITIAL WT. = 70.50g			INITIAL WT. = 125.644g		
	354		42	45						
ON	250		60	60	59.08	83.81	19.37	.001	0	100
ON	177		80	80	4.98	7.06	12.31	.006	.01	99.99
ON	125		115	120	2.73	3.88	5.25	30.41	24.20	75.79
ON	88		170	170	1.86	2.64	2.61	52.50	41.79	34.00
ON	63		250	230	1.28	1.82	0.79	19.77	15.73	18.27
ON	44		325	325	0.10	0.13	0.66	11.19	8.91	9.36
ON	PAN				0.46(1)	0.66		11.76(1)	9.36	
				Totals,	70.50	100		125.64		
					(1) INCLUDES 0.07g LOST			(1) INCLUDES LOSS OF 0.13g		

DATA

Total Shot Weight

Split Sample Yes

No

Sample Weight ________ gms.

Remarks

Figure 5. Sieve analysis for Dominion Ti-6Al-4V and Penn Ti-5Al-2.5Sn.

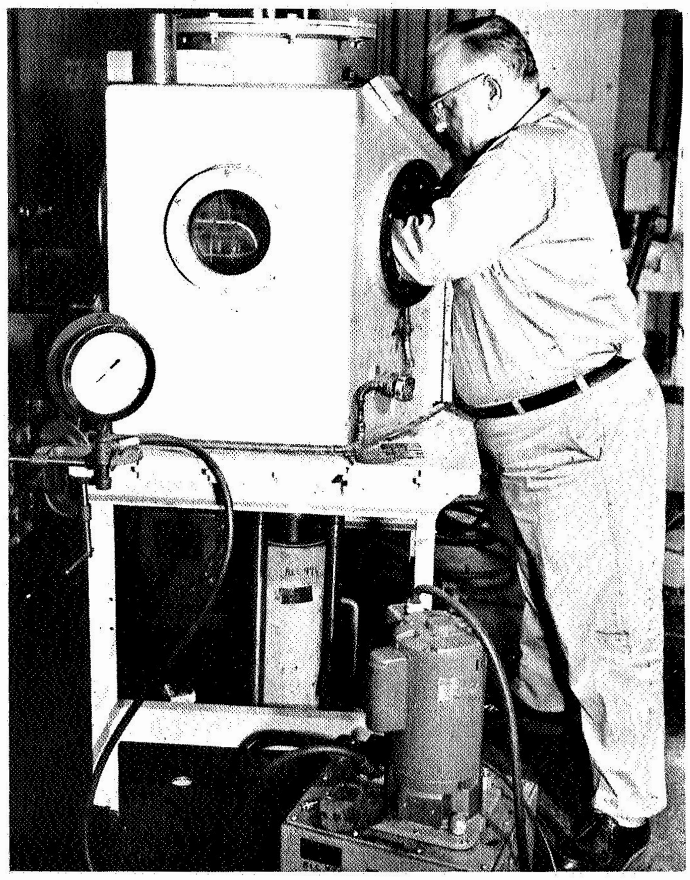


Figure 6. Inert-gas glove box, showing electro-hydraulic oil pump in foreground and compacting piston under box.

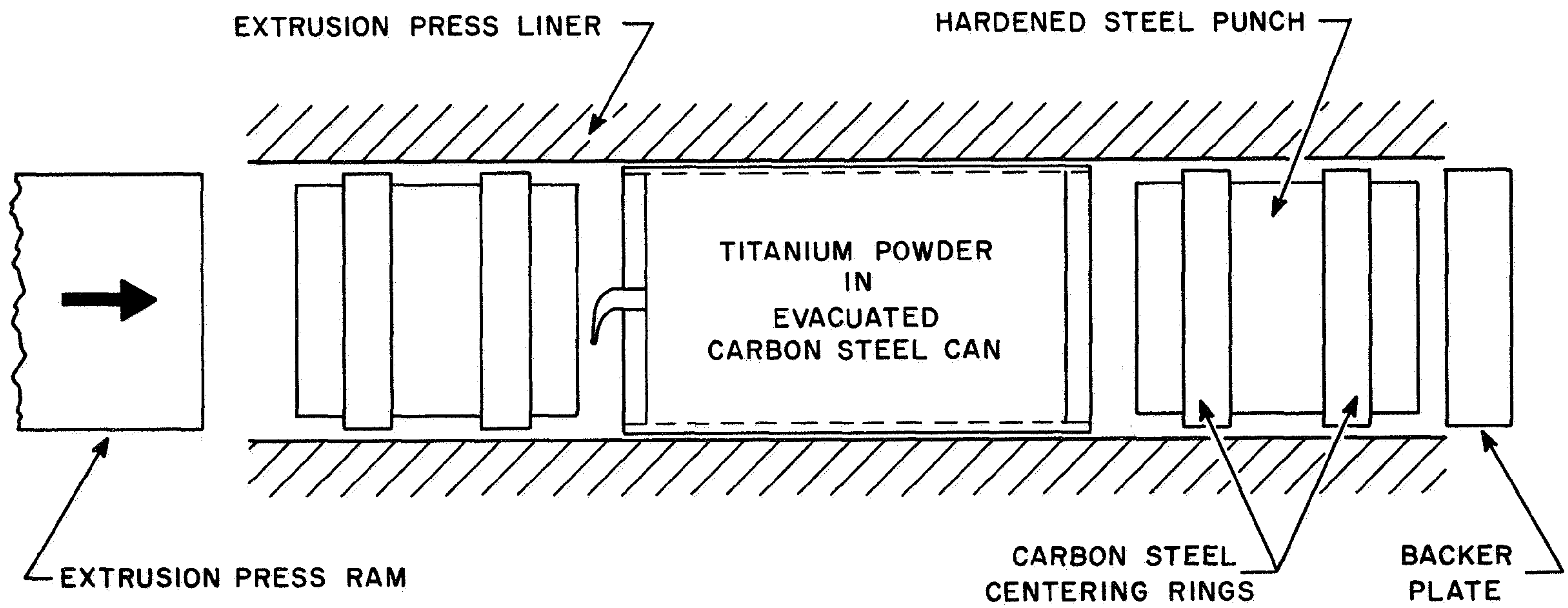


Figure 7. Press setup for hot pressing titanium alloy powder.

RA 3344

An additional set of billets (series 100, 200,[1] etc.) was prepared from powder which was cold pressed in air, rather than under an inert gas cover. These billets were then evacuated, sealed-off, and hot pressed at 1650°F, 75 tsi, except for No. 200, which was pressed at 100 tsi. The purpose of preparing these compacts was to determine the degree of interstitial contamination that would occur as a result of performing the initial filling and pressing operations in air, rather than in an inert atmosphere.

When the billets had cooled to room temperature, they were machined on all surfaces to a depth of 0.025 inch below the titanium-steel interface. The density of each compact was then determined from measurements of weight and physical dimensions (Tables II, III). These data show:

1. None of the Dominion Ti-6Al-4V powder compacts achieved the 98 percent cutoff density established before the start of the program. This situation is explainable in view of this powder's very high interstitial content, which would increase the difficulty of plastically deforming the powder particles.

2. No powder could be compacted to 98 percent density at 50 tsi, 1450°F. Pressures of 75 and 100 tsi were sufficient to produce compacts of over 99 percent density at 1450, 1650, and 1850°F.

It is assumed that the density fractions greater than unity, e.g., 100.5 percent for sample 17 (Numec MA, 1850°F, 50 tsi) and 100.3 percent for sample 56, are due merely to differences in specific chemistry[2] for the powders from which these compacts were made.

A series of hardness determinations were then made along the side, i.e., parallel to the pressing direction, and across the face of each cylinder. The average of these values for each compact over 98 percent dense is listed in Table IV. It can be seen that the softest Ti-5Al-2.5Sn compacts are those made by Nuclear Metals (NM). The Numec hydride-dehydride process (Numec Hyd) and the Nuclear Metals Rotating Electrode Process produced Ti-6Al-4V powder compacts of approximately equal softness. The hardest compacts are those made from the Penn Nuclear Ti-5Al-2.5 Sn powder.

All of the 1850°F/100 tsi compacts were then sectioned as indicated in Figure 8, providing samples for chemistry, heat treating, metallography, and tensile testing. The choice of this set of samples, rather than a set

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[1] The first digit in the sample identification scheme refers to the alloy and powder manufacturer, as listed in Tables II and III. For the samples prepared under inert atmosphere, the second digit refers to the temperatures and pressures indicated in the tables. The samples prepared in air were all compacted under the same conditions (except as noted below) and therefore are undifferentiated with respect to the final digit.

[2] E.g., 5.84 percent aluminum and 4.19 percent vanadium, rather than exactly 5.00 percent aluminum and 4.00 percent vanadium.

TABLE II.— DENSITY OF HOT COMPACTED TITANIUM ALLOY POWDER BILLETS, g/cc

Temperature	1450°F			1650°F			1850°F			1650°F
Pressure (tsi)	50	75	100	50	75	100	50	75	100	75
Reference No.	1	2	3	4	5	6	7	8	9	100[a]
1. MA 6-4	4.256	4.429	4.429	4.435	4.449	4.444	4.448	4.460	4.443	4.451
2. REP 5-2.5 . . .	--	4.405	4.445	4.299	4.438	4.444	4.446	4.447	4.450	4.441[b]
3. REP 6-4	4.286	4.393	4.397	4.397	4.419	4.418	4.411	4.418	4.417	4.411
4. Coreduced 6-4 .	4.038	4.211	4.238	4.180	4.244	4.259	4.256	4.275	4.272	4.237
5. Fluid-energy 5-2.5	4.276	4.442	4.442	4.386	4.438	4.463	4.453	4.452	4.452	4.430
6. Hyd 6-4	4.265	4.392	4.409[c]	4.393	4.411	4.415	4.416	4.456	4.416	4.425
7. Hyd 5-2.5 . . .	4.236	4.422	4.440	4.306	4.433	4.443	4.459	4.440	4.444	4.414
8. MA 5-2.5 . . .	4.261	4.434	4.450	4.340	4.418	4.432	4.452	4.448	4.413	4.448

[a]The samples were handled in air, rather than inert gas, before evacuation and sealing.
[b]Compact 200 - pressed at 100 tsi instead of 75 tsi.
[c]Compact 63 pressed at 1850°F, 100 tsi (duplicating 69).

Legend:

MA - NUMEC Mechanically Attrited Powder
REP - Nuclear Metals Rotating Electrode Powder
Coreduced - Dominion Magnesium Co-reduced Powder
Fluid-energy - Penn Nuclear Gas Attrited Powder
Hyd - NUMEC Hydride-Dehydride Powder

TABLE III.– PERCENT THEORETICAL DENSITY OF HOT COMPACTED TITANIUM ALLOY POWDER BILLETS[a]

Temperature	1450°F			1650°F			1850°F			1650°F
Pressure (tsi)	50	75	100	50	75	100	50	75	100	75
Reference No.	1	2	3	4	5	6	7	8	9	100[b]
1. MA 6-4	96.2	100.1	100.1	100.2	100.6	100.4	100.5	100.8	100.4	100.6
2. REP 5-2.5	--	99.0	99.9	96.6	99.7	99.9	99.9	99.9	100.0	99.8[c]
3. REP 6-4	96.9	99.3	99.4	99.4	99.9	99.9	99.7	99.9	99.8	99.7
4. Coreduced 6-4 . . .	91.3	95.2	95.8	94.5	95.9	96.3	96.2	96.6	96.6	95.8
5. Fluid-energy 5-2.5	96.1	99.8	99.8	98.6	99.7	100.3	100.1	100.0	100.0	99.6
6. Hyd 6-4	96.4	99.3	99.7[d]	99.3	99.7	99.8	99.8	100.7	99.8	100.0
7. Hyd 5-2.5	95.2	99.4	99.8	96.3	99.6	99.8	100.2	99.8	99.9	99.2
8. MA 5-2.5	95.8	99.6	100.0	97.5	99.3	99.6	100.0	100.0	99.2	100.0
Compacts over 98% T.D.:	0	6	6	4	6	6	6	6	6	6

[a]Based on a density of 4.424 g/cc for Ti-6Al-4V and 4.450 g/cc for Ti-5Al-2.5Sn.
[b]These samples were handled in air, rather than inert gas, before evacuation and sealing.
[c]Compact 200 pressed at 100 tsi instead of 75 tsi.
[d]Compact 63 pressed at 1850°F, 100 tsi (duplicating 69).

Legend:

MA - NUMEC Mechanically Attrited Powder
REP - Nuclear Metals Rotating Electrode Powder
Coreduced - Dominion Magnesium Co-reduced Powder
Fluid-energy - Penn Nuclear Gas Attrited Powder
Hyd - NUMEC Hydride-Dehydride Powder

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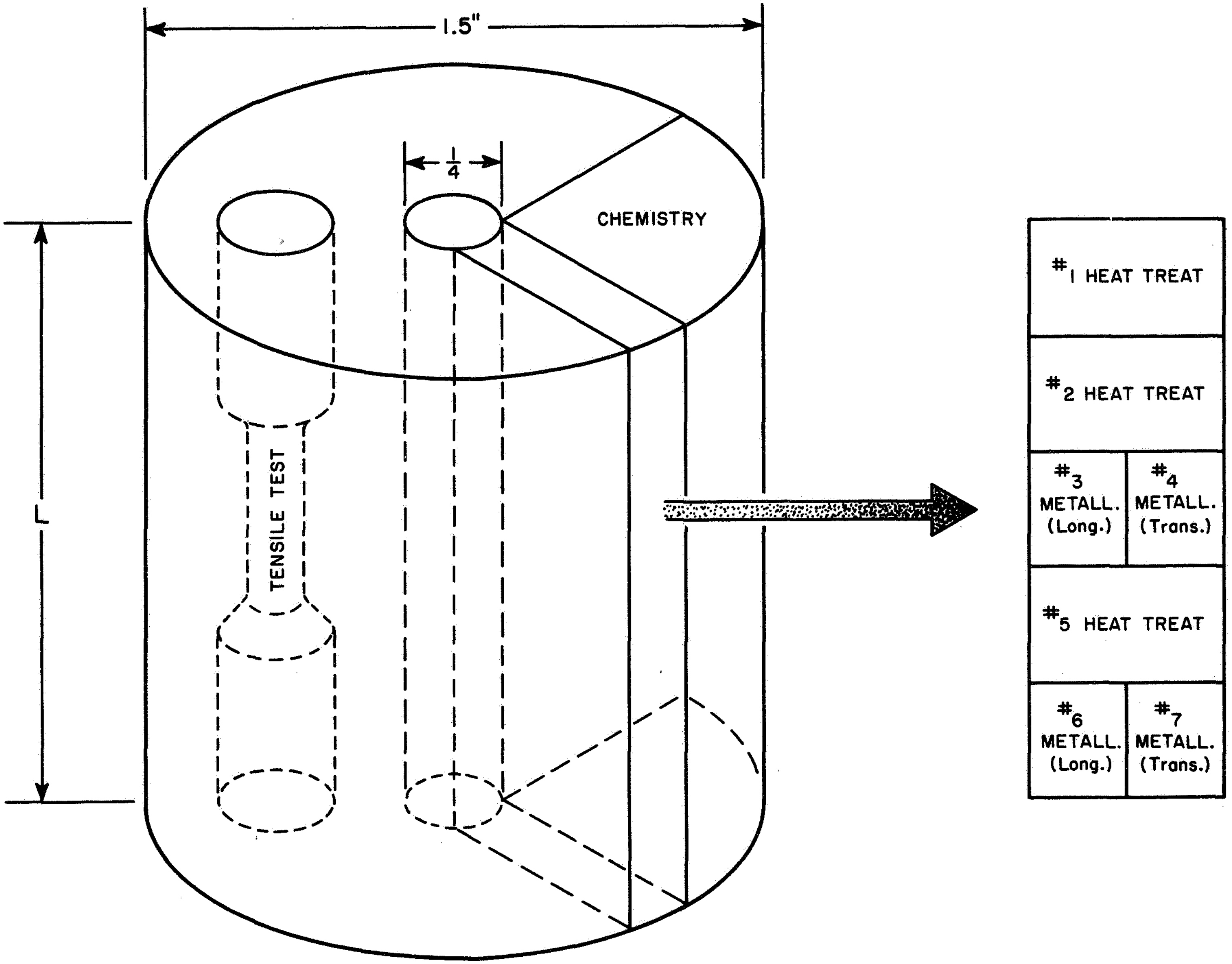


Figure 8. Sectioning of hot pressed billet for test samples.

compacted at a lower temperature or pressure, was made on the theory that properties obtained from these samples would be equal to or better than those in any other set. Samples from other sets were subsequently used in the heat treatment study described below.

Tables V and VI contain the results of chemical analyses performed on the 1850°F/100 tsi compacts. Only the REP samples have low enough oxygen contents to be considered in the ELI category. In all other cases, the oxygen contents are higher than the approximately 1200 ppm established as the ELI limit. For all powders, oxygen content increased during further processing. Table VI charts the progression of oxygen contamination during the conversion of these samples from bar to powder to pressed compact. The oxygen content for the REP powders and most of the Numec powders is low enough to be included in the commercial range for ELI (REP) or standard grade (Numec) titanium. Therefore, the lower-than-standard values for tensile ductility cannot be entirely attributable to oxygen content, but are also related to the poorer forming characteristics of the higher interstitial content, hence harder, powders.

High oxygen powders are difficult to form because the oxygen in them is not uniformly distributed but is instead present in a heavy concentration on the surface of the powder particles. Surface oxygen levels can therefore be considerably higher than the nominal oxygen content for the powder. The high-oxygen surface layer is quite hard and impedes plastic deformation and surface welding of the powder particles. The direct consequence of the hardened particle surface layer is a lowering of bond strength between powder particles, and hence lower mechanical properties for compacts in the as-pressed condition. High temperature annealing treatments can offset this condition somewhat by diffusing oxygen away from the particle surface.

Photomicrographs of the as-pressed compacts are presented in Figure 9. Prior particle boundaries are visible in some of the samples. The structure of the Ti-6Al-4V samples is predominently that of the acicular alpha phase in a framework of prior beta grain boundaries. The Ti-5Al-2.5Sn alloy is single-phase, and the structures observed are those of the alpha phase.

In Table VII are presented the results of a series of annealing treatments performed on 1850°F/100 tsi samples to separate the contributions of sample chemistry and pressing stresses on sample hardness. The heat treatments seem not to have affected the hardness of any sample to a noticeable degree. This may be due to the fact that all heat treatments (which were based on published recommended annealing cycles) took place below the temperature at which the samples were pressed. A more marked response, with a greater difference in hardness between samples, might have been obtained if we had tested samples which had been pressed at 1450°F or 1650°F.

TABLE V.— CHEMICAL ANALYSIS OF HOT PRESSED TITANIUM ALLOY COMPACTS

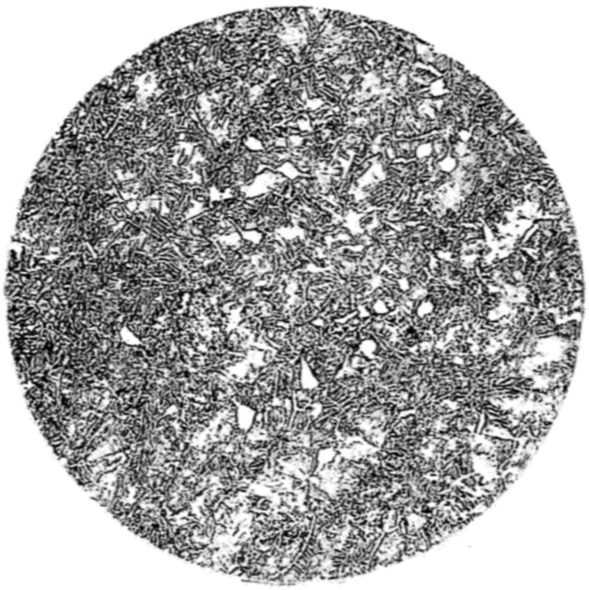
Element:	C (ppm)	O (ppm)	H (ppm)	N (ppm)	Al (%)	V (%)	Sn (%)	Fe (%)
Sample								
Ti-6Al-4V								
MA	650	1710	144	239	5.99	4.17	--	0.19
REP	200	900	90	234	6.04	4.30	--	0.13
Hyd	210	1570	210	148	5.75	4.31	--	0.14
MA, loaded in air . . .	--	1890	--	158	--	--	--	--
REP, loaded in air . .	--	750	--	110	--	--	--	--
Hyd, loaded in air . .	--	3430	--	89	--	--	--	--
Ti-5Al-2.5Sn								
REP	255	980	78	189	5.06	--	2.38	0.21
Fluid energy	640	3530	183	591	5.44	--	2.31	0.31
Hyd	150	3620	52	208	5.03	--	2.38	0.06
MA	500	1640	258	155	6.33	--	[a]	0.14
REP, loaded in air . .	--	845	--	67	--	--	--	--
Fluid energy, loaded in air	--	[b]	--	520	--	--	--	--
Hyd, loaded in air . .	--	2090	--	340	--	--	--	--
MA, loaded in air . . .	--	1755	--	380	--	--	--	--

[a] Incorrect analysis.
[b] 1400-4700.

TABLE VI.– OXYGEN PICKUP DURING FABRICATION OF HOT PRESSED TITANIUM COMPACTS

Sample	Oxygen Content (ppm)		
	Starting Bar	Powder	Hot Pressed Compact
Ti-6Al-4V			
MA	700	1560	1710
REP	700	750	900
Coreduced	---	8100	---
Hyd	700	1300	1570
Ti-5Al-2.5Sn			
REP	950*	750*	980
Fluid energy	---	2800	3530
Hyd	740	1380	3620
MA	740	1220	1640

*The powder has not "lost" 200 ppm oxygen. The figures merely indicate a heterogeneity in the starting stock such that the portion of bar used for making the REP powder contained less oxygen than the portion used for the mill analysis.

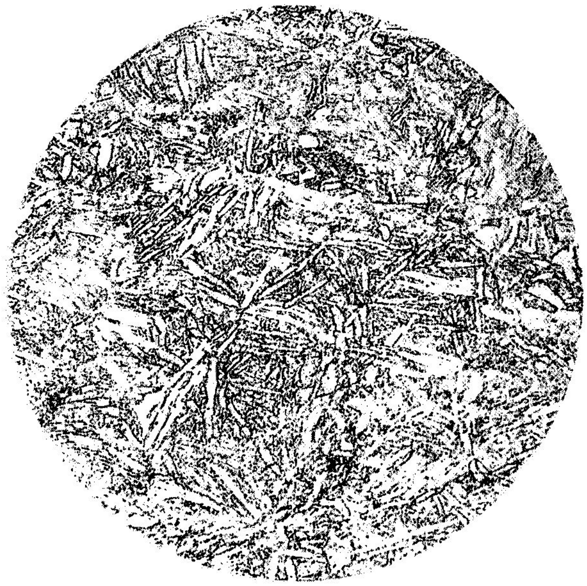


Mechanically attrited

Hydride-dehydride

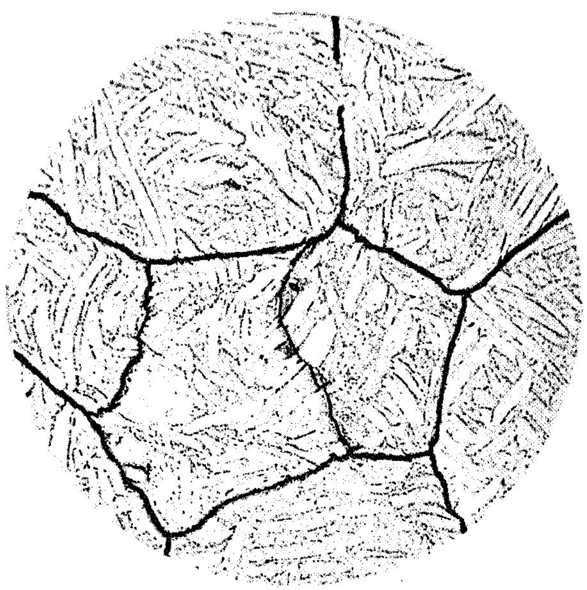
REP

Figure 9a. As-pressed Ti-6Al-4V compacts, 250X, etched.

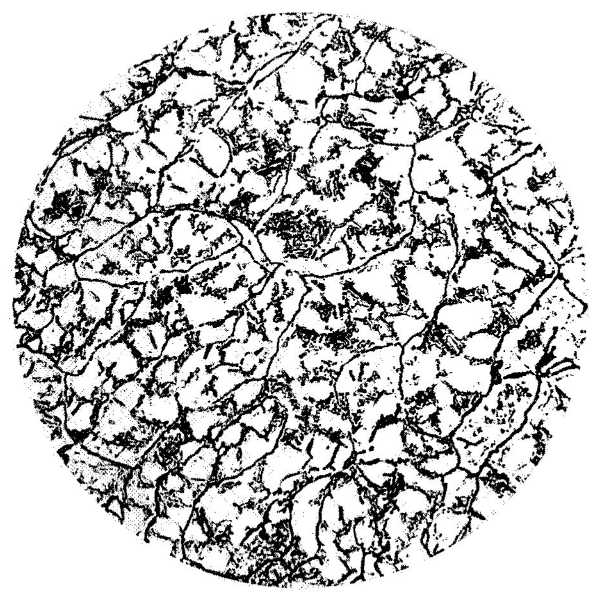


Mechanically attritted

Hydride-Dehydride



REP



Fluid-energy milled

Figure 9b. As-pressed Ti-5Al-2.5Sn compacts, 250X, etched.

TABLE VII.— SAMPLE HARDNESS (R_C) AS A FUNCTION OF HEAT TREATMENT

Ti-6Al-4V												
Sample:	MA			REP			Hydride					
	1	2	5	1	2	5	1	2	5			
As hot pressed[a]	33.6	35.3	35.1	30.1	29.6	31.1	33.3	34.0	34.5			
1300°F/4 hr/WQ[b]	34.3	--	--	30.4	--	--	34.5	--	--			
1425°F/3 hr/WQ	--	35.6	--	--	32.0	--	--	33.2	--			
1550°F/2 hr/WQ	--	--	36.3	--	--	31.3	--	--	34.3			
Ti-5Al-2.5Sn												
Sample:	REP			Fluid energy			Hydride			MA		
	1	2	5	1	2	5	1	2	5	1	2	5
As hot pressed	29.2	29.5	29.0	38.7	40.8	41.8	28.7	28.8	29.3	34.0	34.6	34.1
1325°F/2 hr/AC[c]	31.5	--	--	39.0	--	--	29.6	--	--	36.3	--	--
1440°F/1 hr/AC	--	29.2	--	--	41.2	--	--	27.8	--	--	34.0	--
1550°F/1/2 hr/AC	--	--	29.3	--	--	46.3	--	--	28.2	--	--	33.5

[a] All samples pressed at 1850°F/100 tsi.
[b] Water quenched.
[c] Air cooled.

Tensile properties for the as-pressed compacts are listed in Table VIII. It should be noted that in both groups of compacts -- those in which powder-filling was performed in helium and those filled in air -- the billets were evacuated before being sealed and heated for pressing. From these data we can note a rough correlation between the hardness data in Table VII and the elongation values presented here. It can be seen that the compacts made from the fluid-energy milled powder have high hardness and a correspondingly low level of tensile ductility. The REP and the hydride powders produced softer compacts which yielded higher values of tensile elongation.

The compacts originally filled in air rather than in helium seem in general to reflect a decrease in ductility as a result of this procedure. In the Ti-5Al-2.5Sn alloy, both the REP and the hydride specimens show this effect, as does the REP Ti-6Al-4V specimen. It appears that the higher purity powders are the most vulnerable to oxygen contamination when processed in air.

A comparison between the mechanical property data presented in Table VIII and MIL-T-9047 shows that none of the samples exhibited the minimum tensile elongation required by the specification. A series of heat treatments was therefore performed on the compacts made from the REP powder. This family of compacts was chosen because it possessed the highest purity and therefore offered the greatest potential in achieving high values of ductility and fracture toughness.

The results of these heat treatments are tabulated in Tables IX and X. Figures 11 and 12 illustrate microstructures associated with the heat treated samples.

Examination of the tensile results for the Ti-6Al-4V alloy shows that tensile ductility, as measured by elongation and reduction in area, increases with an increase in compacting and heat treatment temperature and time. The effect of the heat treatments would therefore appear to be that of enhancing the disappearance, by diffusion, of the prior particle boundaries. This effect is shown very clearly in Figures 11a and 11b, in which samples compacted at 1650°F/75 tsi were annealed for two and four hours at 1650°F.

As the annealing temperatures are increased, further evidence of particle-boundary elimination is offered by the increase in grain size which occurs. While the samples annealed at 2200°F possess adequate ductility, we see that tensile strength has begun to fall off (as a result of greatly increased grain size) as compared to the as-pressed condition. There is a large difference in grain size between sample 11 (Figure 11c), annealed at 2200°F, and sample 25 (Figure 11d), which was treated at 1775°F for a similar period of time. The microstructure shown in Figure 11c is one of acicular alpha and prior beta grain boundaries, obtained by heating entirely in the beta phase field (the bounary between the alpha-plus-beta and the beta phase fields lies at 1820°F for Ti-6Al-4V). Sample 25, annealed at 1775°F, was therefore heated at the high end of the alpha-plus-beta field. The structure illustrated is that of primary alpha (white regions) and transformed beta (acicular alpha). The properties obtained by heating at this temperature offer a good balance between tensile strength and ductility.

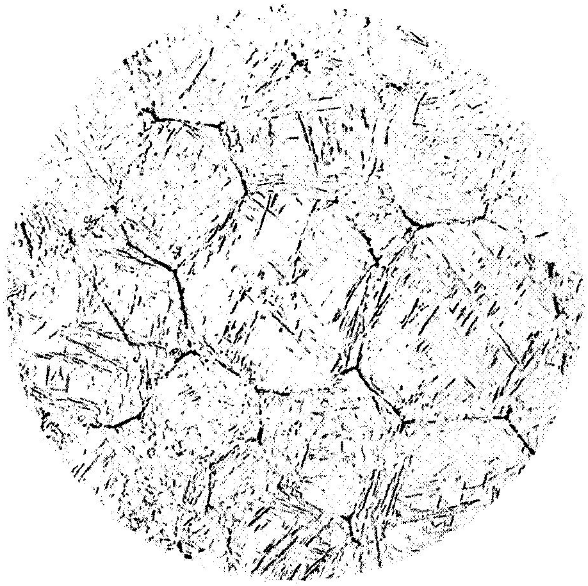
TABLE VIII.— TENSILE PROPERTIES OF TITANIUM ALLOY POWDER COMPACTS

Sample	Billet Cans Filled in Helium Hot Pressed at 1850°F/100 tsi			Billet Cans Filled in Air Hot Pressed at 1650°F/75 tsi		
	UTS (ksi)	0.2% YS (ksi)	Elong. (%)	UTS (ksi)	0.2% YS (ksi)	Elong. (%)
<u>Ti-6Al-4V</u>						
Mechanically attrited	144.8	135.9	1.5	147.5	142.4	1.25
REP	145.1	134.1	7.5	145.6	136.4	2.0
Hyd	149.3	141.3	2.0	153.8	145.8	2.5
MIL Spec.[a], Ti-6Al-4V, Ann.	130	120	10	---	---	---
" " Ti-6Al-4V, ELI, Ann. . .	120	110	10	---	---	---
<u>Ti-5Al-2.5Sn</u>						
REP	130.7	130.7	4.0	44.5	---	0
Fluid energy	129.9	---	0	85.4	---	0
Hyd	126.3	120.3	5.5	21.2	---	0
Mechanically attrited	92.8	---	0	131.7	129.7	2
MIL Spec.[(a)], Ti-5Al-2.5Sn, Ann. . . .	115	110	10	---	---	---
" " Ti-5Al-2.5Sn, ELI, Ann. .	105	100	10	---	---	---

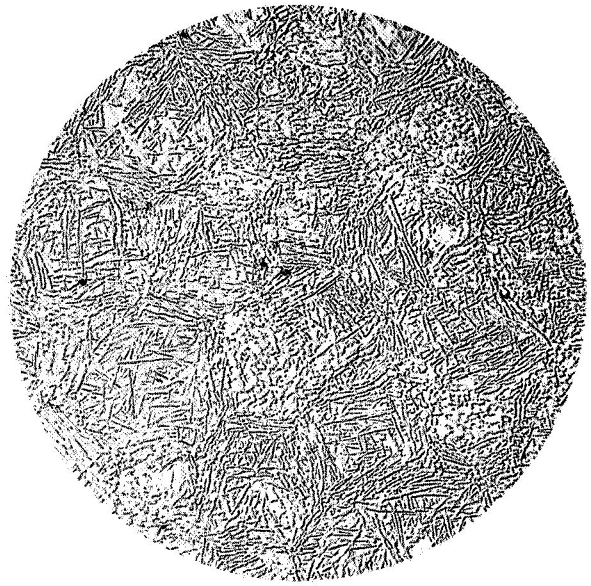
[a]MIL-T-9047D.

TABLE X.— MECHANICAL PROPERTIES OF REP Ti-5Al-2.5Sn, ELI TITANIUM ALLOY COMPACTS AFTER VARIOUS HEAT TREATMENTS

No.	Compacting Conditions Pressure (tsi)	Compacting Conditions Temp. (°F)	Heat Treatment	U.T.S. (ksi)	0.2 Y.S. (ksi)	% Elong. 1" G.L.	% R.A.	True Fracture Stress (ksi)
1	100	1650	1550/4/AC	120.7	114.5	6.5	16.8	135.5
2	100	1650	1550/4/AC	119.2	112.6	9.0	22.0	136.9
3	100	1650	2200/4/AC	100.2	96.5	3.8	29.0	112.3
4	100	1650	2200/4/AC	102.4	96.6	5.1	30.6	107.7
5	100	1850	1850/4/AC	126.6	116.4	13.2	23.4	158.7
6	100	1850	1850/4/AC	127.1	116.4	15.2	42.5	184.3
7	100	1450	1850/4/AC	123.2	113.6	7.7	7.9	139.5
8	100	1450	"	123.8	113.2	10.8	14.2	156.3
9	100	1850	1700/4/AC	122.4	113.0	13.8	26.4	186.1
10	50	1650	1775/4/AC	126.0	113.1	19.1	41.9	180.5
11	75	1650	"	122.5	115.1	11.3	25.8	153.0
12	75	1850	"	128.0	118.5	14.8	44.5	189.5
13	75	1850	"	125.0	116.0	13.7	40.9	177.8
14	75	1850	1850/4/AC	127.0	116.5	13.5	38.8	174.5
15	75	1850	"	127.5	116.9	13.0	36.0	173.4

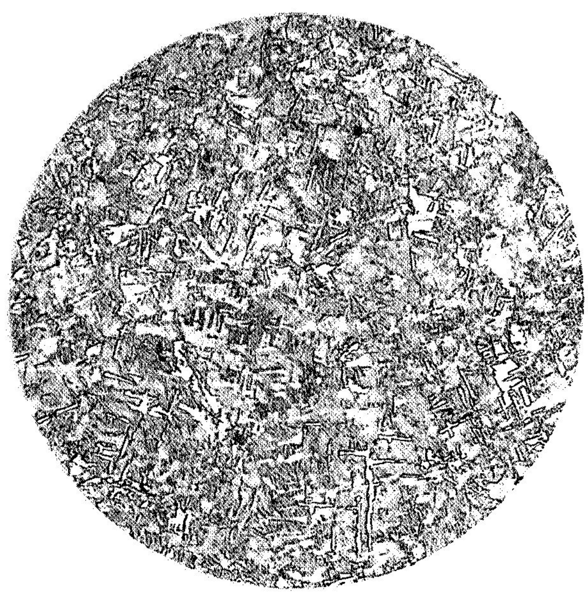


a. Pressed: 1650°F/75 tsi
Heat treated: 1650°F/2/WQ



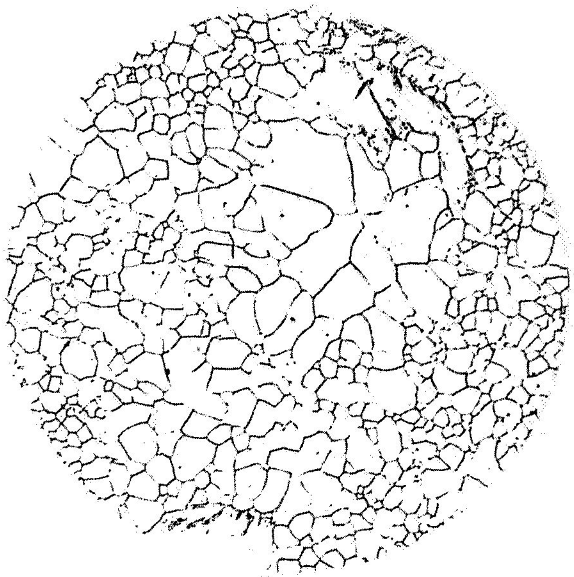
b. Pressed: 1650°F/75 tsi
Heat treated: 1650°F/4/WQ

c. Pressed: 1650°F/75/tsi
Heat treated: 2200°F/4/AC

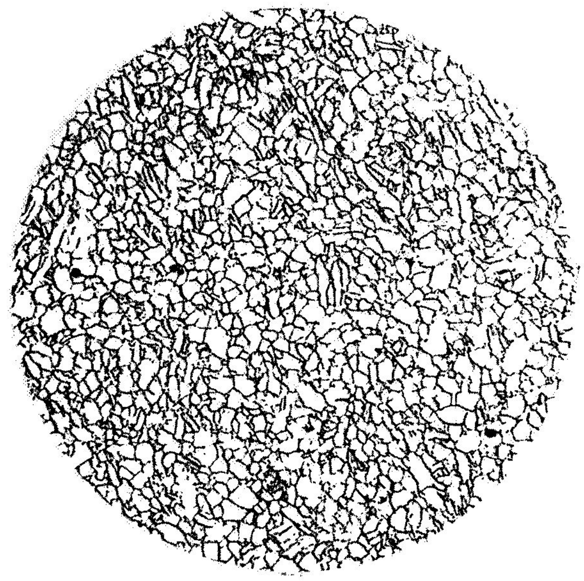


d. Pressed: 1850°F/75 tsi
Heat treated: 1775°F/4/AC + 1300°F/2/AC

Figure 11. REP Ti-6Al-4V, pressed and heat treated as indicated. Transverse sections, 100X, etched.



a. Pressed: 1650°F/100 tsi
Heat treated: 1550°F/4/AC



b. Pressed: 1850°F/100 tsi
Heat treated: 1850°F/4/AC

c. Pressed: 1850°F/75 tsi
Heat treated: 1775°F/4/AC

Figure 12. REP Ti-5Al-2.5Sn, pressed and heat treated as indicated. Transverse sections, 100X, etched.

Higher strengths can be realized by subjecting the sample to a solution treatment, as was done for samples 6 - 10. The aging treatment, which was omitted for these samples, would have raised strengths even further, with a probable further loss in ductility. However, this procedure would be undesirable for several reasons:

1. Mechanical properties based upon water quenching small samples would be difficult to reproduce in objects of large size, such as large forged valve and pump bodies, which are of ultimate interest here.

2. The solution treatment sacrifices ductility for strength, whereas it would be most desirable to maximize ductility.

3. The lower tensile strengths which are produced in samples tested at room temperature by annealing rather than by solution treating will be more than made up in the cryogenic strengthening which will occur naturally when samples are tested at very low temperatures.

Ti-5Al-2.5Sn, being a single-phase alloy, cannot be strengthened by heat treatment. The objective of any heat treatment for this alloy is therefore only to relieve working stresses, or (as in the present case) to diffuse away particle boundaries. Figure 12 illustrates the structure of Ti-5Al-2.5Sn tensile specimens. Sample 16, which was compacted at 1850^{o}F/75 tsi, was subsequently annealed for four hours at 1775^{o}F. The micro-structure consists of alpha grains in a plate form.

The result of the heat treatment - tensile test experiments was to develop a suitable treatment producing adequate tensile strength along with good ductility in hot pressed REP titanium alloy powders. Both the hot pressing techniques and the post-pressing heat treatments are applicable to larger bodies, e.g., the 6-inch by 6-inch by 3-inch blocks, with no anticipated loss in properties.

Production and Testing of 6-Inch by 6-Inch by 3-Inch Blocks

Block Preparation

Ti-6Al-4V and Ti-5Al-2.5Sn electrode stock was extruded from the large forged bars. These extruded rods, approximately 1-1/8 inches in diameter, were then machined to electrode size and converted to powder via the Rotating Electrode Process. Chemical analysis and other pertinent data for the two alloy powders appear in Table XI.

TABLE XI.—DESCRIPTION OF REP Ti-6Al-4V ELI AND Ti-5Al-2.5Sn ELI POWDERS

			Ti-6Al-4V	Ti-5Al-2.5Sn
1. Weight of powder produced (lb)			52	36
2. Flow rate (sec)			21.8	24.6
3. Apparent density (g/cc)			2.68	2.84
Apparent density (% theoretical)			60.5	63.8
4. Particle size distribution:				
	Mesh (Tyler)	Microns		
Percent on screen:	24	710	-----	2.0
	32	500	-----	14.6
	42	354	8.5	38.2
	60	250	31.8	30.3
	80	177	40.0	11.6
	115	125	13.2	3.11
	170	88	4.9	.8
	250	63	.9	.1
	325	44	.3	0
Pass:	325	44	.1	0

5. Chemical analysis:

Element (%)	Al	V	Sn	Fe	C (ppm)	O (ppm)	N (ppm)	H (ppm)
Ti-6-4 powder	6.4	4.29	----	.133	196	730	175	58
Ti-6-4 forged bar	6.31	4.27	----	.110	270	700	70	1b
Ti-5-2.5 powder	5.22	----	2.43	.040	40	510	268	34
Ti-5-2.5 forged bar	5.46	----	2.80	.020	60	740	140	6

A set of rectangular tool steel punches was made up to fit a 3-1/4-inch by 6-1/4-inch by 22-inch hot pressing die. The die cavity was made up of four tool steel sections that were shrunk into an 8-1/4-inch diameter extrusion container in the 1400-ton extrusion press. The tooling arrangement consisted of a rear punch with a 3-1/8-inch by 6-1/8-inch face which was placed in the liner ahead of the 10-inch long can containing the titanium powder. (At an apparent density of 60.5 to 63.8 percent of theoretical, a powder "length" of approximately 10 inches is required to produce a fully dense block 6 inches long.) After the hot powder can is placed in the die cavity, is is compressed between the rear punch and a front punch which is carried forward by the extrusion ram. This tooling arrangement is illustrated schematically in Figure 13. Figure 14 illustrates the rectangular inserts in the extrusion container; the punch arrangement is shown in Figure 15.

Two 16 gauge carbon steel cans, 3-1/8 inches by 6-1/8 inches by 10 inches long, were filled with powder, welded, evacuated, and sealed off. Each can contained about 18-3/4 pounds of powder. The sealed cans were heated to 1850°F over a 3- hour period, held at temperature for 1 additional hour, and compacted at 73 tons per square inch. The temperature of the compacting tools was 850°F.

The compacted powder blocks were then heat treated for 4 hours at 1775°F followed by 2 hours at 1300°F. After the steel cans were dissolved in nitric acid, the blocks were weighed and measured:

	Ti-6Al-4V	Ti-5Al-2.5Sn
Length (In pressing direction), inch	6-1/4	6-1/4
Width, inch	3-1/8	3-1/8
Height, inch	6-1/8	6-1/8
Weight, lbs.	18.65	18.8
Density (Displacement Method) g/cc	4.42	4.45
Density, % Theoretical	100%	100%

The appearance of the blocks after removal of the steel cans is shown in Figure 16. Tensile and fracture toughness specimen blanks were then machined from the blocks, following the sectioning diagram shown in Figure 17.

Specimens were also machined from 6-inch lengths of the forged Ti-6Al-4V and Ti-5Al-2.5Sn starting stock. The arrangement of the specimens in these bars is shown in Figures 18 and 19.

All specimens were individually identified according to the scheme shown in the sectioning diagrams. They were then shipped to the Whittaker Research and Development Division in San Diego for final machining and testing.

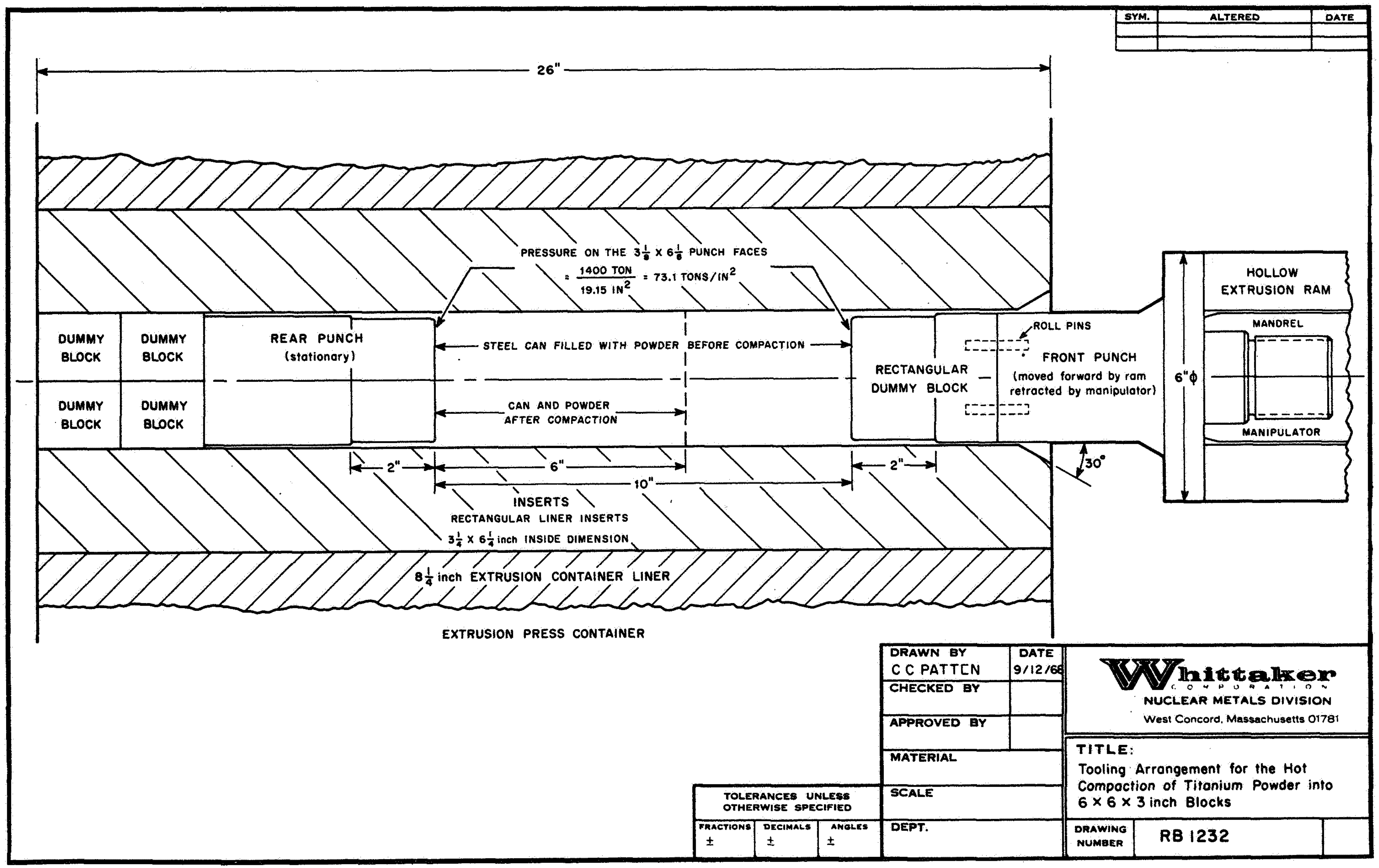


Figure 13. Arrangement of powder compaction tooling in the 1400-ton extrusion press.

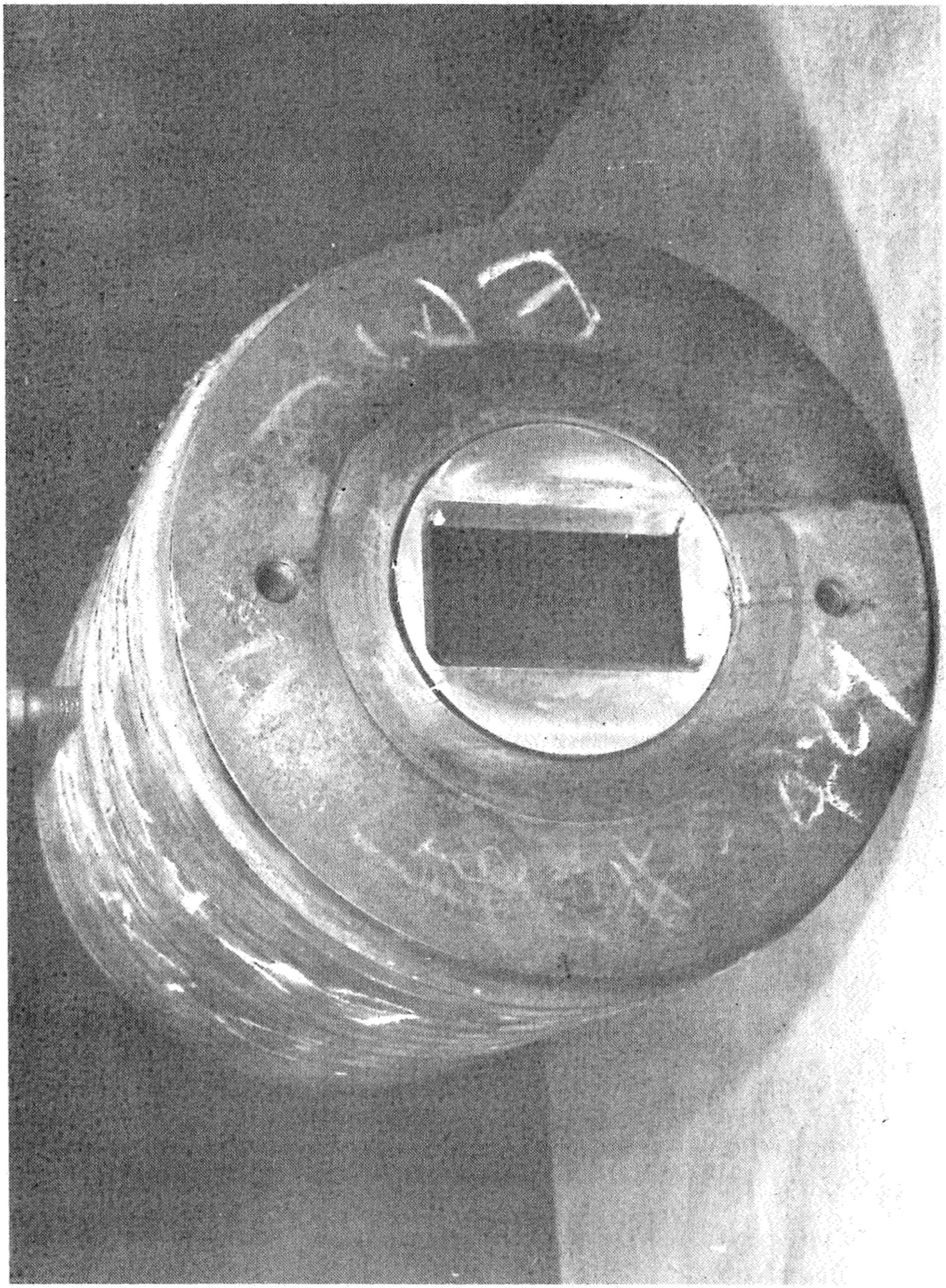


Figure 14. The 6-1/4 by 3-1/4 by 22 inch inserts in extrusion press container.

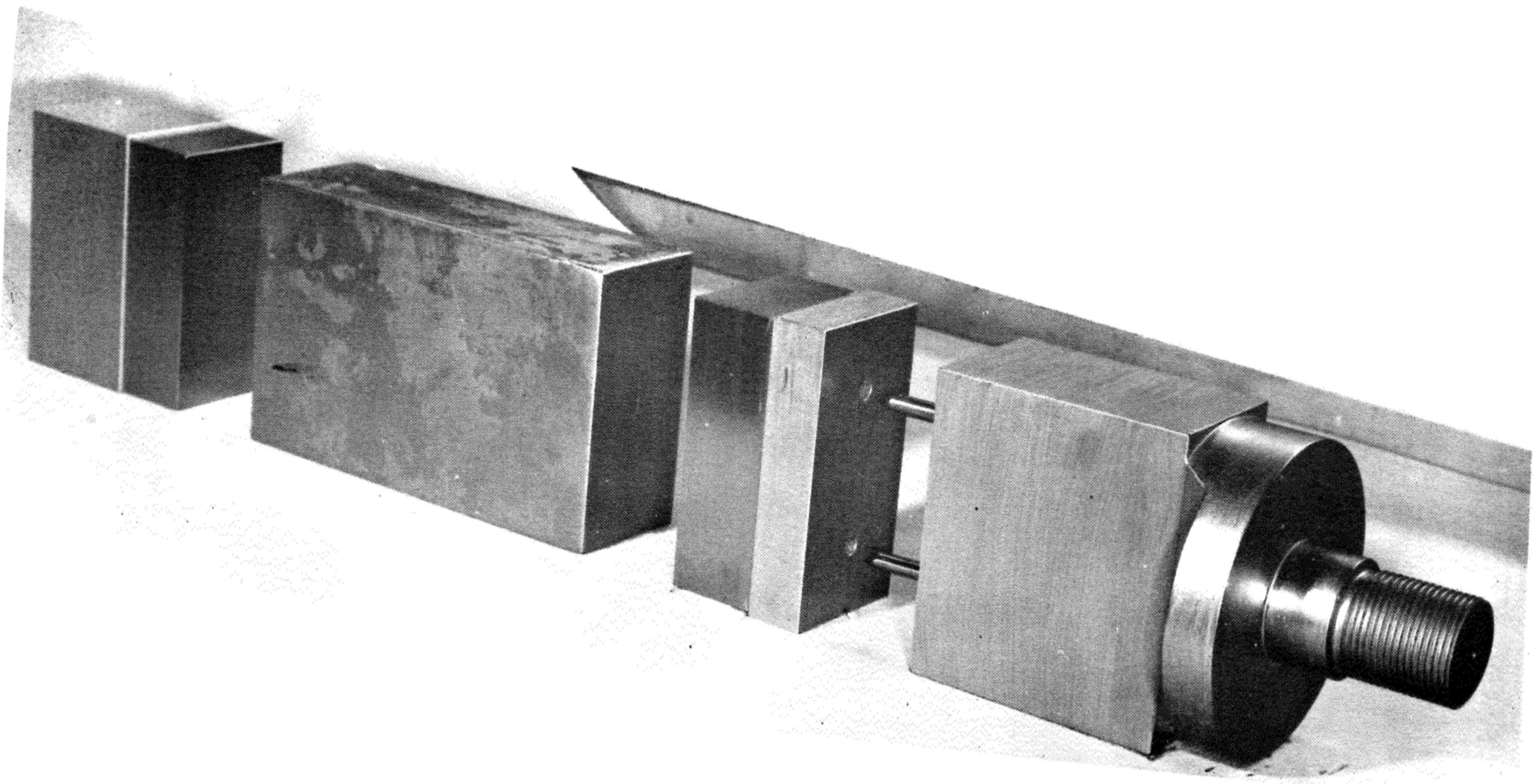


Figure 15. Arrangement of tooling for compaction of titanium powder.

Left to right:

1. Rear punch
2. Steel can, 6-1/8 by 3-1/8 by 10 inches, containing titanium powder.
3. Dummy block (front punch)
4. Front punch holder

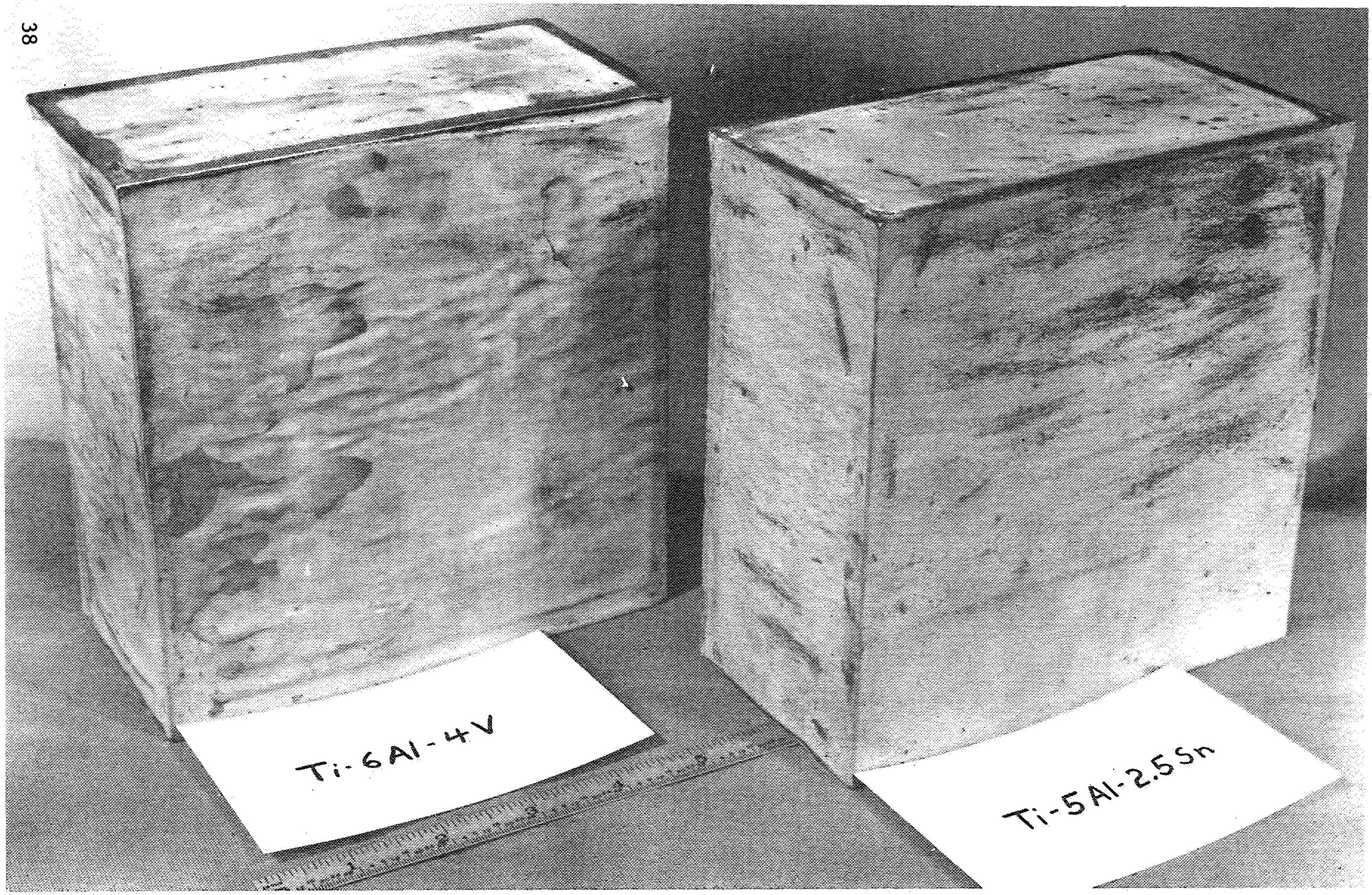
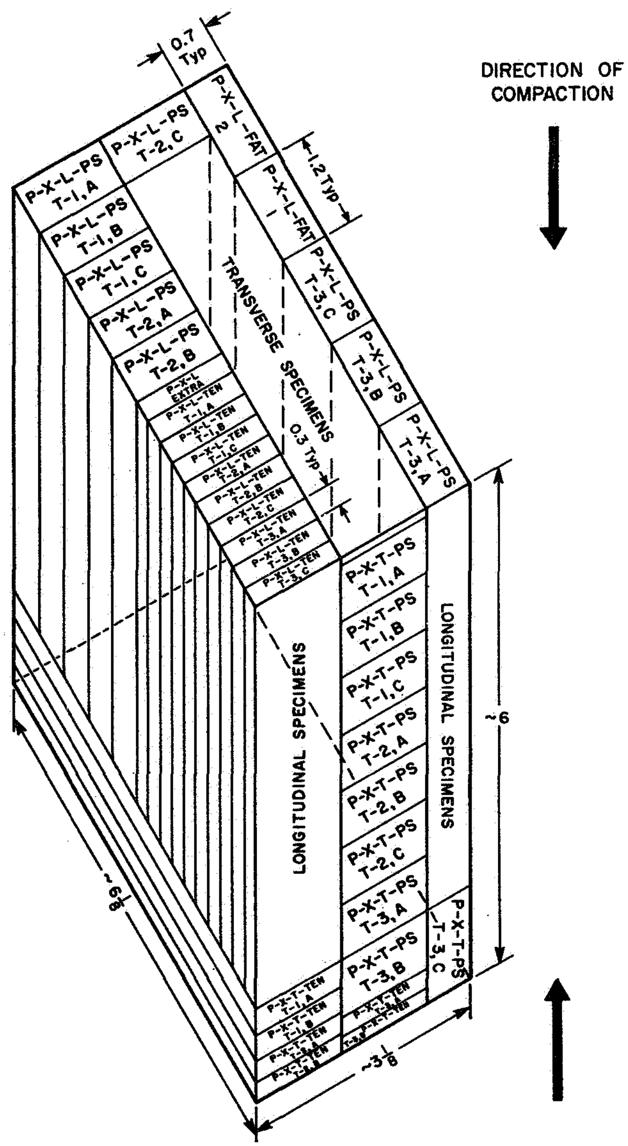


Figure 16. Hot pressed titanium blocks after removal of steel cans.



LEGEND

P = HOT PRESSED POWDER BLOCK

X = 6, FOR Ti-6Al-4V
5, FOR Ti-5Al-2.5Sn

L = LONGITUDINAL (parallel to pressing direction)

T = TRANSVERSE

T-1 = ROOM TEMPERATURE

T-2 = 320°F

T-3 = 423°F

SPECIMEN BLANK SIZE

TENSILE : 5" X 1" X 0.1"

PLAIN STRAIN : 5" X 1" X 0.5"

FATIGUE : 5" X 1" X 0.5"

DIMENSIONS INDICATED ON DRAWING ARE FOR LAYOUT PURPOSES ONLY

RC 3365

SYM.	ALTERED	DATE

DRAWN BY	DATE
CHECKED BY G/F	6-13-66
APPROVED BY	
MATERIAL	
SCALE	
DEPT.	

TOLERANCES UNLESS OTHERWISE SPECIFIED

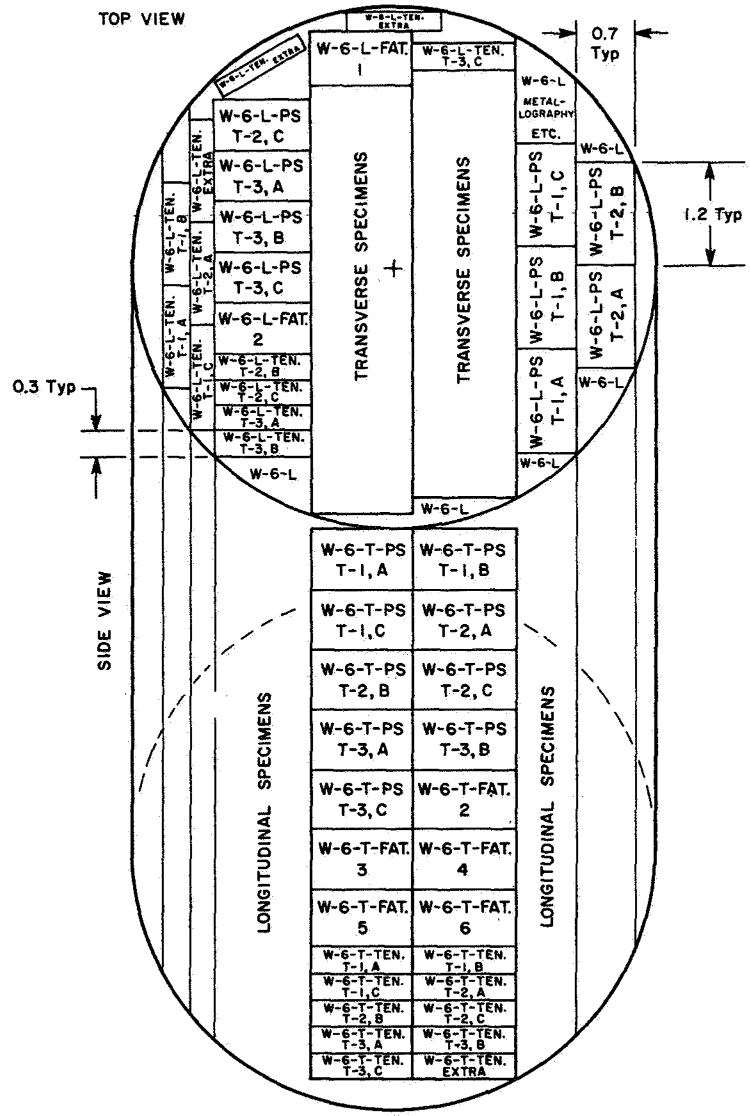
FRACTIONS	DECIMALS	ANGLES
±	±	±

NUCLEAR METALS, INC.
CONCORD, MASSACHUSETTS
RESEARCH DEVELOPMENT

TITLE:

DRAWING NUMBER RC 3365

Figure 17. Arrangement of specimen blanks in hot pressed titanium alloy blocks



LEGEND

W = WROUGHT
6 = Ti-6Al-4V
L = LONGITUDINAL DIRECTION
T = TRANSVERSE DIRECTION
T-1 = ROOM TEMPERATURE
T-2 = −320°F
T-3 = −423°F
PS = PLAIN STRAIN SPECIMEN
TEN. = TENSILE SPECIMEN
FAT. = FATIGUE TRIAL SPECIMEN

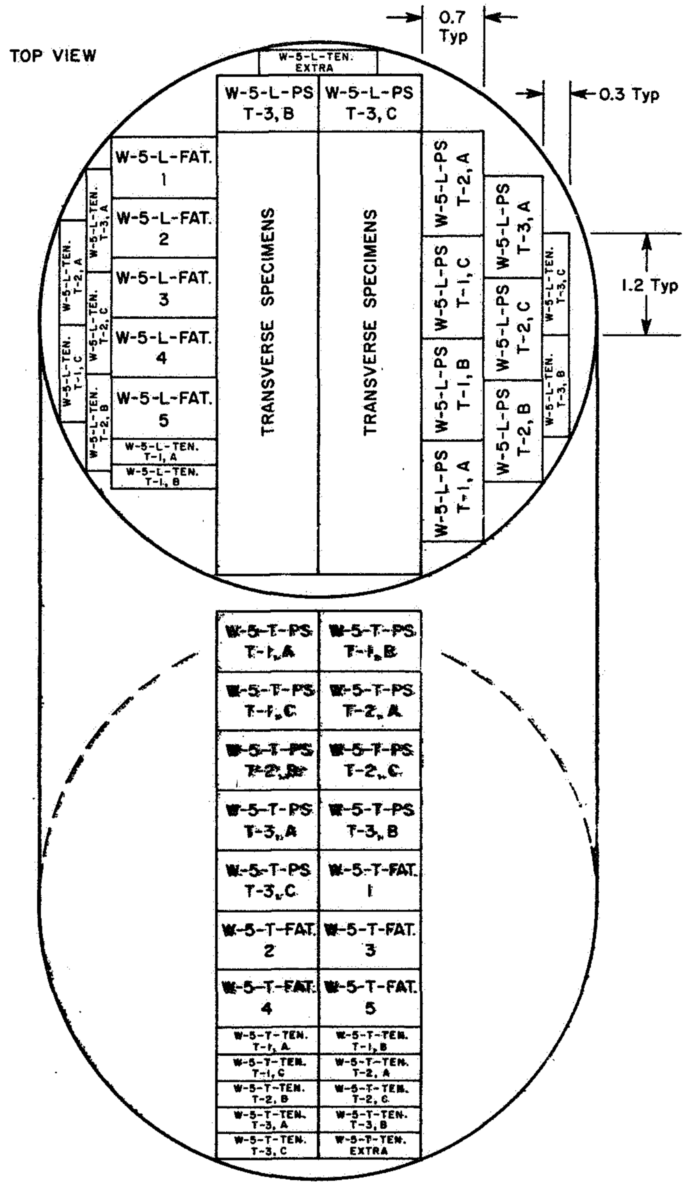
EXAMPLE : W-6-L-PS-T-1, A = WROUGHT Ti-6Al-4V, LONGITUDINAL, PLAIN STRAIN ROOM TEMPERATURE SPECIMEN A

SPECIMEN BLANK SIZE

TENSILE : 5" X 1" X 0.1"
PLAIN STRAIN : 5" X 1" X 0.5"
FATIGUE : 5" X 1" X 0.5"

DIMENSIONS INDICATED ON DRAWING ARE FOR LAYOUT PURPOSES ONLY

Figure 18. Arrangement of specimen blanks in 6-inch forged Ti-6Al-4V bar.



LEGEND

W = WROUGHT
5 = Ti-5Al-2.5 Sn
L = LONGITUDINAL DIRECTION
T = TRANSVERSE DIRECTION
PS = PLAIN STRAIN FRACTURE TOUGHNESS SPECIMEN
TEN. = TENSILE SPECIMEN
FAT. = FATIGUE SPECIMEN
T-1 = ROOM TEMPERATURE
T-2 = -320°F
T-3 = -423°F

SPECIMEN BLANK SIZE

TENSILE : 5" X 1" X 0.1"
PLAIN STRAIN : 5" X 1" X 0.5"
FATIGUE : 5" X 1" X 0.5"

DIMENSIONS INDICATED ON DRAWING ARE FOR LAYOUT PURPOSES ONLY

Figure 19. Arrangment of specimen blanks in 6-1/2-inch forged Ti-5Al-2.5Sn bar.

Tensile Testing

The specimen configuration for the tensile tests is shown in Figure 20. Figure 21, a close-up photograph of the specimen gage section, clearly illustrates the manner in which the tensile specimens were instrumented with strain gages for measurement of elongation, modulus of elasticity, and Poisson's ratio. The surface of the specimens was roughened to increase adherence of the gages. Duplicate gages attached to the reverse side of the specimens were used to cancel any error introduced by possible non-axial loading in the test set-up.

Triplicate tensile tests were performed on longitudinal (parallel to pressing direction, in the case of the powder specimens) and transverse specimens from the powder blocks and the wrought bars. Tests were conducted at room temperature, -320°F (boiling point of nitrogen), and -423°F (boiling point of hydrogen).

Test results are tabulated in Table XII. It can be seen from this tensile data that in almost all cases the powder specimens show properties equivalent to or better than those for the wrought specimens. The Ti-6Al-4V powder samples are stronger than their wrought counterparts at all temperatures, in both the longitudinal and transverse orientation. The ductility of the Ti-6Al-4V powder samples is greater than that of the wrought specimens under all conditions, except at -423°F, where the longitudinal powder samples are slightly inferior.

In the case of the Ti-5Al-2.5Sn alloy, the transverse powder specimens are stronger and more ductile than their wrought counterparts at the three test temperatures. The longitudinal powder specimens, however, show a lower yield strength at -320°C, lower tensile strength at the three test temperatures, and lower overall ductility at room temperature. At -320°F, the powder specimens show greater ductility as measured by reduction in area, but less ductility as measured by specimen elongation. The situation with respect to these two ductility criteria is reversed at -423°F. There are no obvious indications explaining why these longitudinal powder specimens do not show the superior mechanical properties, as compared to the wrought samples, that is evidenced by the other powder specimens.

Portions of the tensile specimens were used for local density determinations, hardness determinations, metallographic examination of structure, (Figure 22), and chemical analysis. All samples were 100 percent dense. Sample hardness ranged from R_c 23.3 to R_c 28 for Ti-5Al-2.5Sn and from R_c 30 to R_c 34.7 for the Ti-6Al-4V samples.

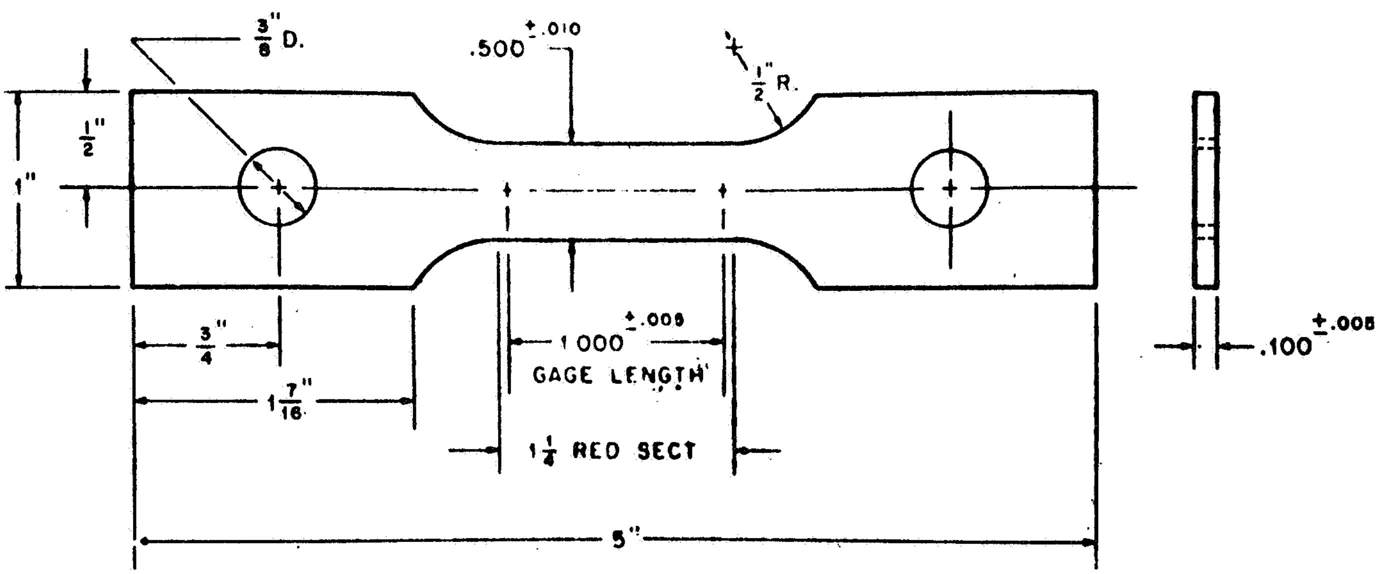


Figure 20. Tensile test specimen.

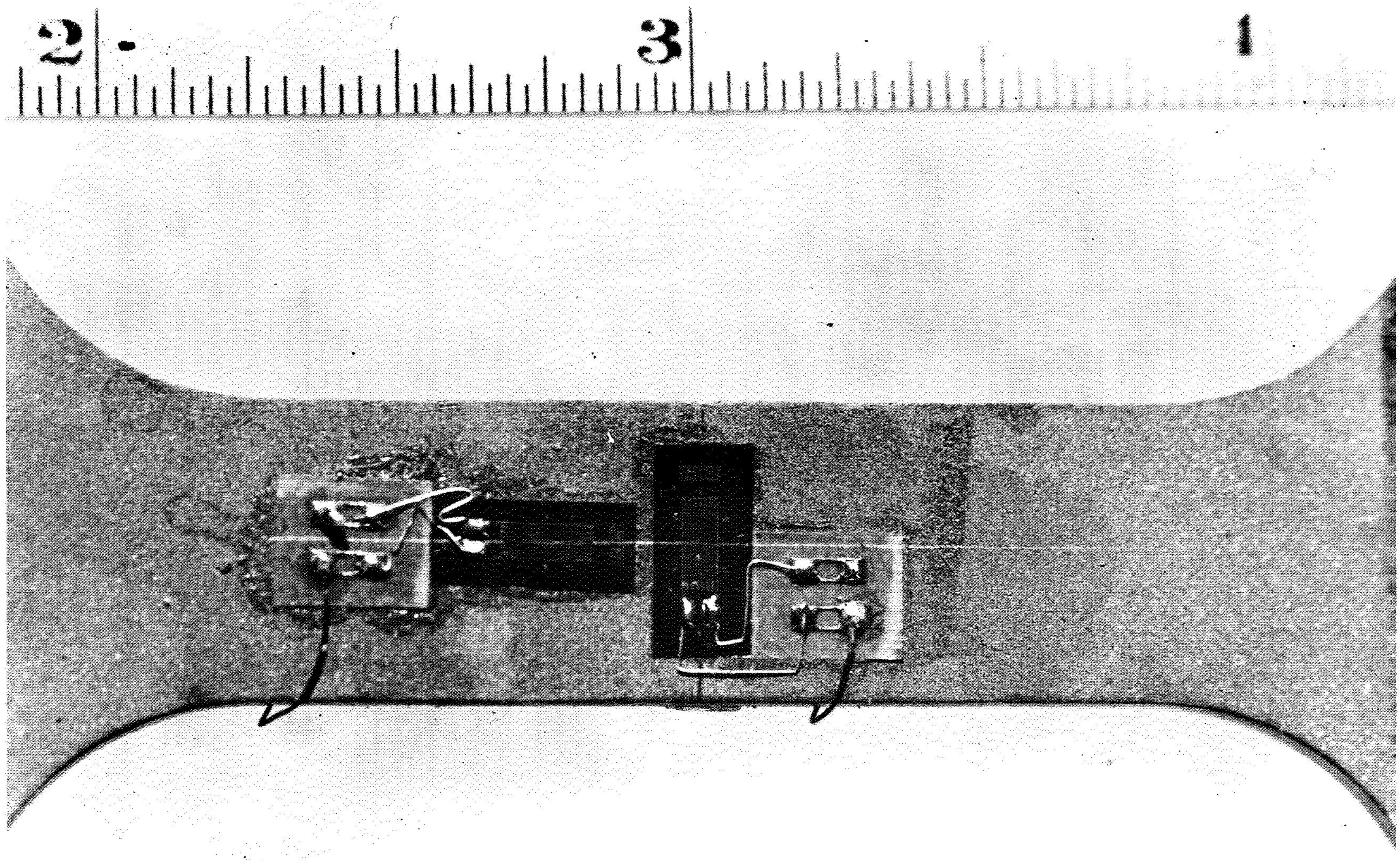


Figure 21. Arrangement of strain gages on tensile specimen.

TABLE XII. TENSILE PROPERTIES[1] OF TWO TITANIUM ALLOYS, IN WROUGHT AND POWDER FORM, AT ROOM AND AT CRYOGENIC TEMPERATURES

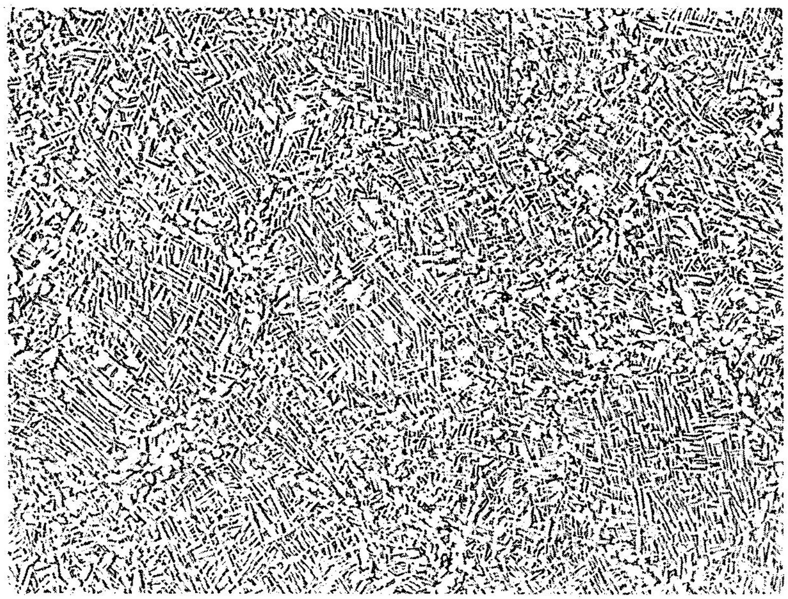
	Ti-6Al-4V, ELI		Ti-5Al-2.5Sn, ELI	
	Wrought[2]	Powder[4]	Wrought[3]	Powder[4]
A. Room Temperature				
Longitudinal				
UTS (ksi)	128	130	118	115
0.2% YS (ksi)	106	114	103	104
Elongation (%)	15.3	18.9	19.3	15.7
RA (%)	29.3	31.3	34.0	27.0
E (psi x 10^6)	15.4	18.0	15.3	17.4
μ	.318	.327	.308	.308
Transverse				
UTS (ksi)	129	132	96.8	113
0.2% YS (ksi)	85.2	122	82.4	99.7
Elongation (%)	10.5	20.2	11.0	20.7
RA (%)	12.0	33.0	28.0	32.1
E (psi x 10^6)	14.1	18.4	15.2	18.4
μ	.278	.319	.298	.310
B. -320°F				
Longitudinal				
UTS (ksi)	202	216	187	178
0.2% YS (ksi)	174	188	170	157
Elongation (%)	12.0	11.7	17.0	10.0
RA (%)	11.3	17.3	16.8	18.6
E (psi x 10^6)	15.4	17.2	18.6	17.7
μ	.353	.306	.341	.287
Transverse				
UTS (ksi)	200	212	171	172
0.2% YS (ksi)	170	189	149	149
Elongation (%)	9.6	10.8	11.3	16.0
RA (%)	11.7	13.0	14.3	18.5
E (psi x 10^6)	18.3	16.1	17.4	17.3
μ	.314	.297	.315	.301

(1) Each value is an average of three tests.
(2) 6-inch annealed forged bar.
(3) 6-1/2-inch annealed forged bar.
(4) Compacted at 1850°F/70 tsi + annealed 1775°F/4 hr/FC + 1300°F/2 hr/AC.

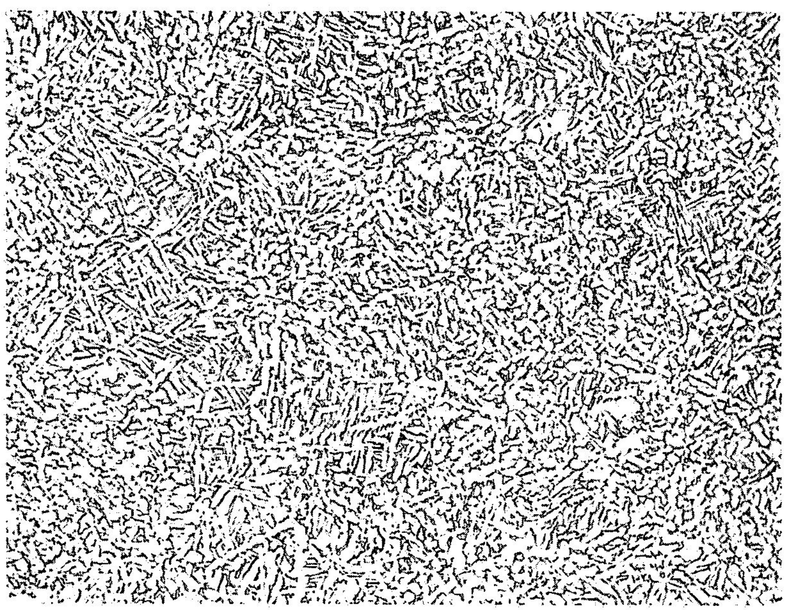
TABLE XII. TENSILE PROPERTIES[1] OF TWO TITANIUM ALLOYS, IN WROUGHT AND POWDER FORM, AT ROOM AND AT CRYOGENIC TEMPERATURES - concluded

	Ti-6Al-4V, ELI		Ti-5Al-2.5Sn, ELI	
	Wrought[2]	Powder[4]	Wrought[3]	Powder[4]
C. <u>-423°F</u>				
Longitudinal				
UTS (ksi)	225	237	214	197
0.2% YS (ksi)	193	197	159	174
Elongation (%)	5.3	3.8	6.8	7.1
RA (%)	12.4	11.6	15.1	13.0
E (psi x 10^6)	18.7	17.6	18.9	20.2
μ	.365	.347	.310	.348
Transverse				
UTS (ksi)	223	237	198	203
0.2% YS (ksi)	166	212	152	160
Elongation (%)	6.9	7.0	8.7	11.0
RA (%)	5.8	10.6	15.3	15.6
E (psi x 10^6)	20.0	17.9	19.8	18.7
μ	.346	.348	.330	.350

(1) Each value is an average of three tests.
(2) 6-inch annealed forged bar.
(3) 6-1/2-inch annealed forged bar.
(4) Compacted at 1850°F/70 tsi + annealed 1775°F/4 hr/FC + 1300°F/2 hr/AC.



Ti-6Al-4V



Ti-5Al-2.5Sn

Figure 22. Microstructure of Ti-6Al-4V and Ti-5Al-2.5Sn powder specimens.

Analyses of samples taken from the tensile specimens compared with the analysis of forged bars and powder indicate that very little change in chemistry had taken place:

Specimen	Percent				Parts Per Million			
	Al	V	Sn	Fe	C	O	H	N
Ti-6-4 Wrought Bar	6.31	4.27	----	0.11	270	700	16	70
Ti-6-4 Powder	6.40	4.29	----	0.13	196	730	80	175
Ti-6-4 H.P. Block	6.09	4.21	----	----	310	1000	71	280
Ti-5-2.5 Wrought Bar	5.46	----	2.80	0.02	60	740	6	140
Ti-5-2.5 Powder	5.22	----	2.43	0.04	40	510	34	268
Ti-5-2.5 H.P. Block	4.87	----	2.36	----	54	640	45	200

Fracture-Toughness Testing

Plain-strain fracture toughness testing of the wrought and powder specimens was performed in general accordance with ASTM STP 410, "Recommended Practice for Plain-Strain Fracture Toughness Testing of High Strength Materials." The procedure was adjusted by a modification of the bend fixture to permit the testing to be performed in the cryostat.

This recommended practice covers a procedure for the determination of the plain-strain fracture toughness, designated K_{IC} (stress x length$^{1/2}$), of high-strength metallic materials by a bend test of a pre-notched and fatigue-cracked rectangular specimen.

The value of K_{IC} so obtained characterizes the susceptibility of a material to unstable crack extension under conditions of high constraint. The plastic deformation of a constrained specimen correspondes to a plain-strain state of stress near the crack front in neutral environments. The K_{IC} value measured by this practice is believed to represent a lower limiting value of fracture toughness.

K_{IC} is a material property which is measured in terms of K_I (stress intensity factor) and is the critical value of K_I which characterizes the susceptibility to unstable crack extension under conditions of high constraint to plastic deformation. K_{IC} of metallic materials increases with the toughness of the materials. Therefore, the specimen size required to obtain valid K_{IC} values increases with the toughness of the material. Although the basic specimen size can be estimated on the basis of the ratio of yield strength to Young's Modulus, the proof of the validity of a test is not known until the test is completed.

As there is no advance assurance that a valid K_{IC} will be determined in a particular test, it is necessary first to calculate a conditional result, K_Q, which involves a construction on the test record. It is then determined whether this result is consistent with the size and yield strength of the specimen.

For a notched and fatigue-cracked rectangular specimen tested in three point bending, the following procedure* is used to calculate K_Q.

(1) The load, P_Q is determined by a construction on the test record in accordance with Paragraph 7.3.1 of the ASTM Recommended Practice.

(2) K_Q is calculated from P_Q as follows:

$$K_Q = \frac{P_Q L}{B w^{3/2}} \left[5.8 \left(\frac{a}{w}\right)^{\frac{1}{2}} - 9.2 \left(\frac{a}{w}\right)^{\frac{3}{2}} + 4.3 \left(\frac{a}{w}\right)^{\frac{5}{2}} - 75.3 \left(\frac{a}{w}\right)^{\frac{7}{2}} + 77.4 \left(\frac{a}{w}\right)^{\frac{9}{2}} \right]$$

where P_Q = load in pounds as determined by construction per Paragraph 7.3.1

B = thickness of specimen in inches

L = one-half the total span length (distance between supports) in inches

w = depth of specimen in inches

a = depth of specimen notch plus fatigue crack in inches

---

*As per "Recommended Practice for Plain Strain Fracture Toughness Testing of Metallic Materials Using a Fatigue Cracked Specimen" prepared by Subcommittee I on Fracture Testing of High Strength Materials, ASTM Committee E-24 (Fracture Testing of Metals), April 1967.

The value of a is determined by measurements made at the most advanced point of the crack front and at each surface of the specimen. The sum of the two surface values plus twice the value at the most advanced point divided by four, is used in the above equation to calculate K_Q.

To facilitate calculation of K_Q, values of the power series given in brackets in the above equation have been determined and plotted against the ratio (a/w). The values for f[a/w] were obtained from the "Recommended Practice." The resultant curve is shown in Figure 23.

For a plain-strain fracture toughness test to be valid (i.e. $K_Q = K_{IC}$), the relationship

$$2.5 \ (K_Q/\sigma_{YS})^2$$

where K_Q = conditional plain-strain fracture toughness

σ_{YS} = .2 percent offset yield strength

must be less than both the specimen thickness B and the depth of the specimen notch plus fatigue crack a (ref. Figure 24). If both B and a are greater than the value obtained from the above relationship, the test is valid and K_Q is equal to K_{IC}. If B and/or a are less than the value determined above, the test is invalid, and it becomes necessary to use a larger specimen -- one in which both B and a are greater than the value determined above -- to obtain valid K_{IC} values.

The specimen design used for the fracture toughness tests is shown in Figure 24. Figure 25 illustrates the position of a test specimen in the test fixture, and Figure 26 shows the room temperature test arrangement.

The results obtained by testing the wrought and powder specimens at three temperatures are presented in Table XIII. Based on the plain-strain criteria discussed above, only the Ti-6Al-4V powder specimens tested at -423°F produced valid results, because these specimens were the only ones in which the value $2.5 \ (K_Q/\sigma_{YS})^2$ was less than both the specimen thickness and the crack length. It is obvious, however, that all the powder specimens yielded lower toughness values than the wrought specimens. The reason for this uniform difference is unknown at present. The explanation probably lies in the relationship between powder processing temperatures and the resultant microstructures; i.e. lower processing temperatures would have yielded finer grain size and a different structure. If, for example, the compacts were pressed below the alpha plus beta transformation temperature, at 1650-1700°F rather than at 1850°F, a primary alpha plus transformed beta microstructure rather than the acicular alpha structure would be obtained. It is quite possible that such a microstructural change would result in superior toughness characteristics. Figure 27 illustrates the fracture surfaces of wrought and powder Ti-5Al-2.5Sn specimens tested at three temperatures. Note the coarse fracture surface of the wrought specimens compared to the powder samples.

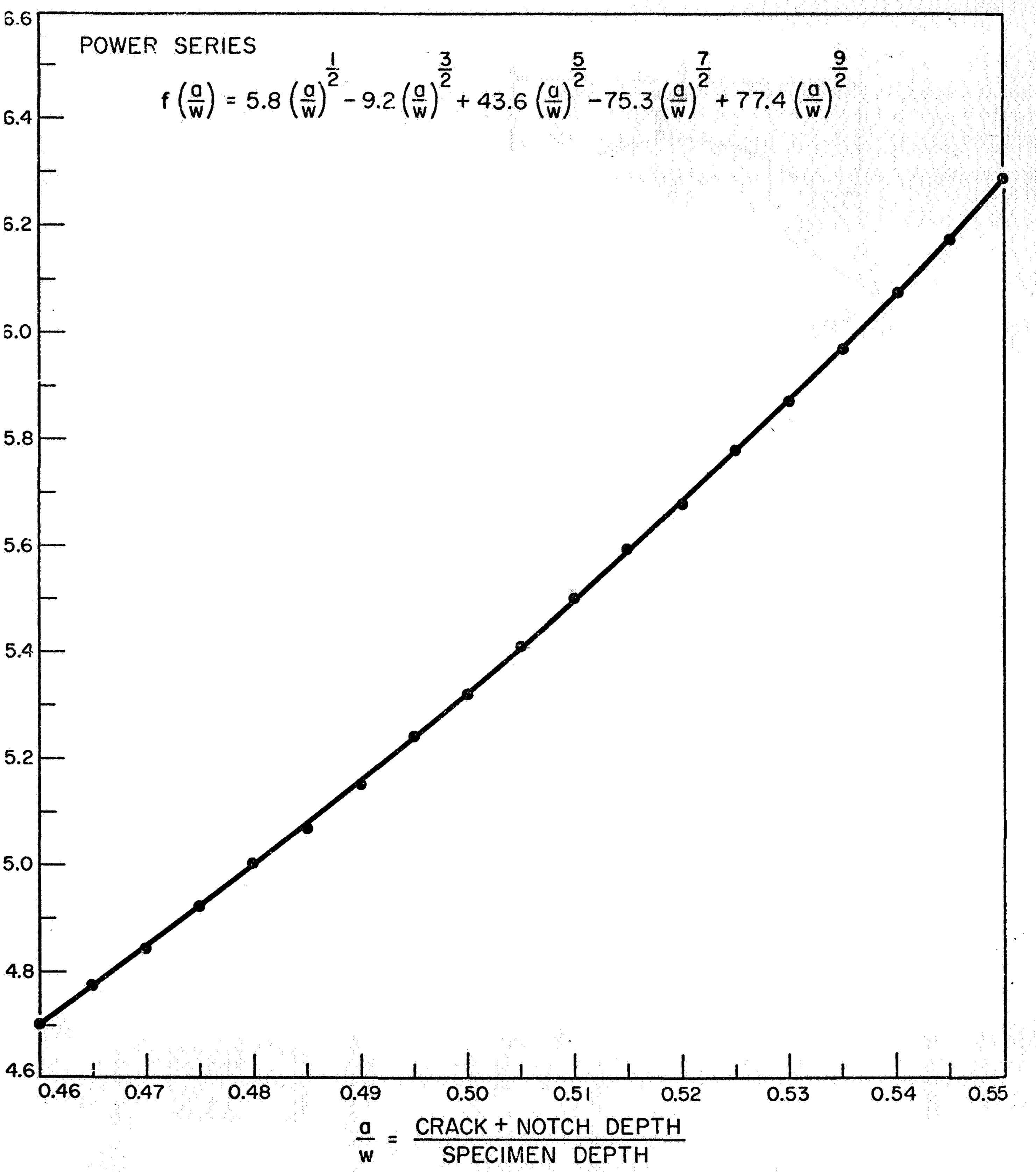


Figure 23 Power Series, f(a/w) vs a/w

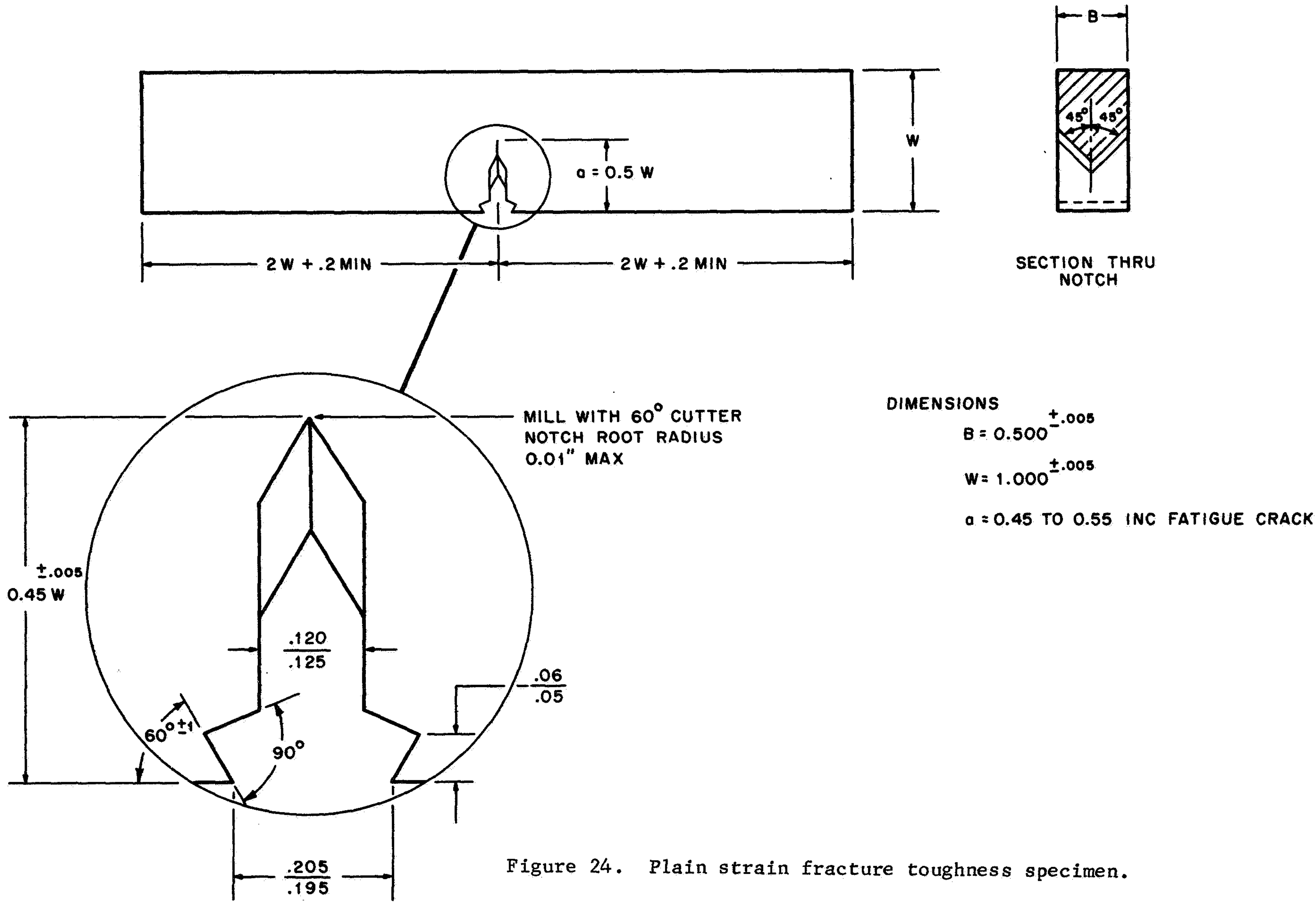


Figure 24. Plain strain fracture toughness specimen.

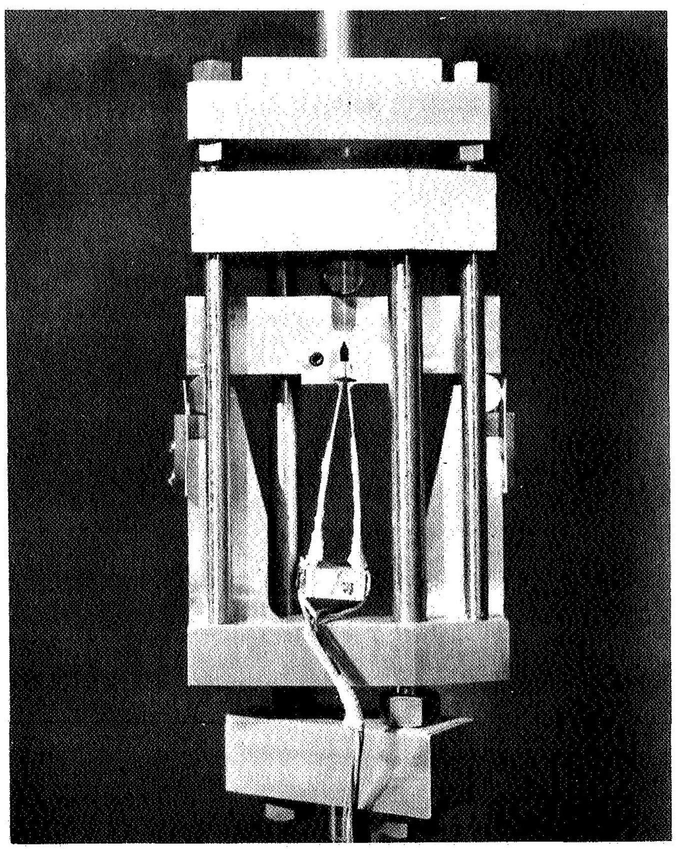


Figure 25. Fracture toughness specimen in test fixture.

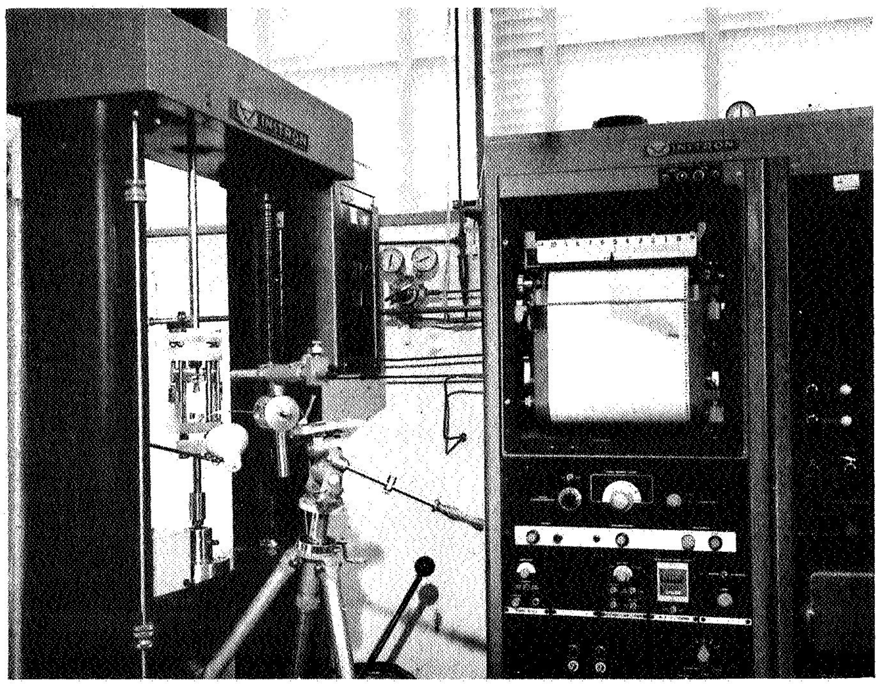


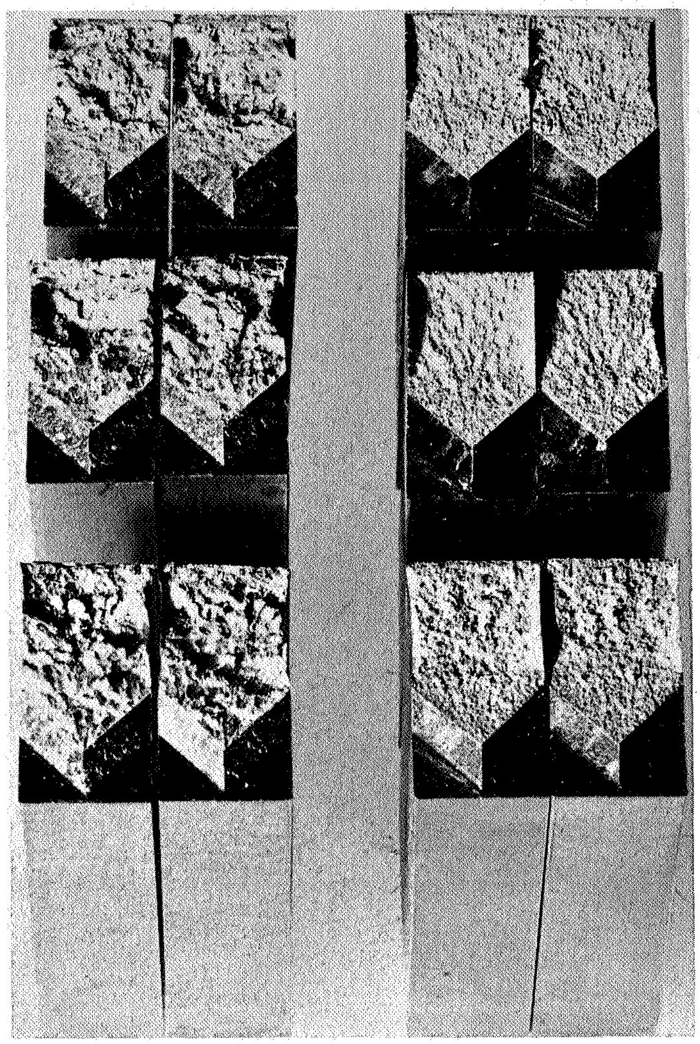
Figure 26. Set-up for plain-strain fracture toughness testing.

TABLE XIII. PLAIN-STRAIN FRACTURE TOUGHNESS FOR TWO TITANIUM ALLOYS, IN WROUGHT AND POWDER FORM

	Ti-6Al-4V, ELI				Ti-5Al-2.5Sn, ELI			
	Wrought		Powder		Wrought		Powder	
	K_Q (psi$\sqrt{in}$)	$f\left(\frac{K_Q}{YS}\right)$* (inches)	K_Q (psi$\sqrt{in}$)	$f\left(\frac{K_Q}{YS}\right)$* (inches)	K_Q (psi$\sqrt{in}$)	$f\left(\frac{K_Q}{YS}\right)$* (inches)	K_Q (psi$\sqrt{in}$)	$f\left(\frac{K_Q}{YS}\right)$* (inches)
A. Room Temp.								
Longitudinal								
A	84,500	1.59	79,000	1.20	84,400	1.59	77,800	1.40
B	90,400	1.82	------	----	-------	----	------	----
C	80,400	1.44	------	----	75,600	1.27	------	----
Transverse								
A	77,500	2.07	74,100	.92	87,600	2.82	78,200	1.56
B	69,200	1.65	------	----	82,700	2.52	------	----
C	------	----	------	----	91,400	3.08	------	----
B. -320°F								
Longitudinal								
A	100,200	.83	86,900	.54**	109,200	1.03	97,000	.96
B	120,200	1.19	------	----	122,700	1.30	------	----
C	-------	----	------	----	101,600	.89	------	----
Transverse								
T A	105,500	.96	82,400	.48***	100,900	1.15	93,600	.99
B	-------	----	------	----	96,400	1.05	------	----
C	-------	----	------	----	86,900	.85	------	----
C. -423°F			K_{IC}					
Longitudinal								
A	92,500	.57**	65,700	.28***	103,600	1.06	82,400	.56
B	106,800	.77	72,100	.33***	107,700	1.15	83,900	.58
C	101,400	.69	69,733	.33***	129,200	1.65	87,000	.62
Transverse								
A	89,800	.73	74,200	.31***	97,400	1.03	79,700	.62
B	88,500	.71	75,600	.32***	90,300	.88	86,700	.73
C	93,500	.79	75,566	.33***	101,600	1.11	83,600	.68

* $f\left(\frac{K_Q}{YS}\right)$ must be less than specimen thickness (**B**) and notch-plus-crack length (a). B and a $\approx$ 0.5 inch.

**Borderline: $f\left(\frac{K_Q}{YS}\right)$ is less than B, but greater than a. ***Valid tests.



-423°F

-320°F

Room Temperature

Wrought

Powder

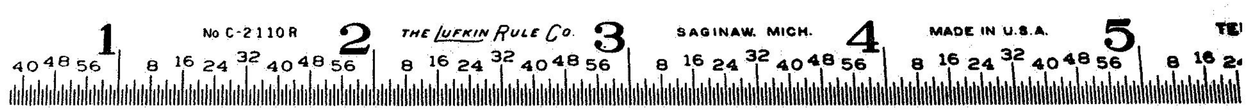


Figure 27. Fracture surface of Ti-5Al-2.5Sn plain-strain specimens.

A calculation has been made, based upon the data in Table XIII, to produce valid test results from Ti-5Al-2.5Sn and Ti-6Al-4V samples. The bases for the calculations are:

(1) A specimen thickness at least 1-1/2 times as large as the value of 2.5 $(K_Q/\sigma_{YS})^2$, as listed in Table XIII.

(2) ASTM specimen size relationships, which dictate a specimen width equal to twice its thickness and a length equal to or greater than four times the specimen width plus 0.4 inch.

The specimen sizes thus obtained are listed in Table XIV. The specimen lengths listed in this table indicate that it would be impractical to test transverse specimens, since a forged bar over three feet in diameter would be required for the Ti-5Al-2.5Sn wrought sample at room temperature, and a compacted powder block over 20 inches wide would be required for the corresponding powder specimen. Such sample sizes are more easily produced from longitudinal specimens, in which case the powder samples could be prepared by hot extrusion.

TABLE XIV. MINIMUM SPECIMEN SIZE TO INSURE PLAIN-STRAIN CONDITIONS IN WROUGHT AND POWDER SPECIMENS OF Ti-6A;-4V (ELI) AND Ti-5Al-2.5Sn (ELI)

Alloy	Temp. (°F)	Direction	$2.5\ (K_Q/YS)^2$ (inches) [from Table XIII]	Thickness* (inches) B	Width (inches) W [=2B]	Length (inches) L [=4W+0.4in,min]
Ti-6Al-4V (wrought)	RT	L	1.8	2.7	5.4	22
		T	2.1	3.2	6.4	26
	-320	L	1.2	1.8	3.6	15
		T	.96	1.44	2.9	12
	-423	L	1.65	2.4	4.8	19
		T	1.11	1.7	3.4	14
Ti-6Al-4V (powder)	RT	L	1.20	1.8	3.6	13
		T	.92	1.4	2.8	12
	-320	L	.54	.81	1.62	7
		T	.48	.72	1.44	7
	-423	L	.33	.50	1.0	5(valid as is)
		T	.33	.50	1.0	5(valid as is)
Ti-5Al-2.5Sn (wrought)	RT	L	1.6	2.4	4.8	19
		T	3.1	4.6	9.2	37
	-320	L	1.3	2.0	4.0	17
		T	1.2	1.8	3.6	15
	-423	L	1.65	2.4	4.8	19
		T	1.11	1.7	3.4	14
Ti-5Al-2.5Sn (powder)	RT	L	1.40	2.1	4.2	18
		T	1.56	2.34	4.7	20
	-320	L	.96	1.44	2.9	12
		T	.99	1.50	3.0	13
	-423	L	.62	.93	1.86	8
		T	.73	1.10	2.2	9.5

$$\text{*Thickness} = 1\text{-}1/2\ \left[2.5\ \left(\frac{K_Q}{YS}\right)^2\right]$$

CONCLUSIONS

This program demonstrated that of the five powder-making processes investigated, only the Rotating Electrode Process (REP) was capable of producing ELI-grade titanium alloy powder. The oxygen content of compacted bodies made from the REP Ti-6Al-4V and Ti-5Al-2.5Sn powders was 1000 ppm or less. In comparison, the compacts made from powder produced by the hydride-dehydride process, the mechanical attriting of chips, fluid-energy (gas) mill attriting of chips, and chemical reduction had oxygen contents ranging from 1600 to over 8000 ppm.

All powders, except those made by chemical reduction, could be compacted to full density at a temperature/pressure combination of 1650°F and 75 tons/inch^2. The chemically reduced powder could not be compacted to over 96.6 percent density at 1850°F and 100 tons/inch^2, the highest temperature/pressure combination investigated.

The as-compacted powder samples possessed only limited ductility, with elongation values ranging from 0 to 7.5 percent. The REP powder samples developed over 10 percent elongation and 40 percent reduction in area after four hour anneals at 1775°F.

Blocks hot-pressed from the spherical REP powders had tensile properties equivalent to or better than those obtained from wrought bar. In particular, the powder Ti-6Al-4V block specimens were stronger and more ductile than the wrought bar samples at all test temperatures, in both longitudinal and transverse directions. The powder Ti-5Al-2.5Sn samples showed this same superiority in the transverse direction only. Longitudinal powder specimens of this alloy were either weaker or less ductile than corresponding wrought samples.

Plain-strain fracture toughness tests showed that the sample size chosen was too small for valid tests to be performed. For specimens as tough as these, it would be necessary to double or triple specimen dimensions to provide plain-strain conditions during testing.

Although valid K_{IC} values were not obtained, a comparison of the data is possible, since both the powder and wrought specimens were of the same size and shape. All the powder specimens exhibit apparent lower toughness than the wrought samples, at all temperatures. The difference in K_Q between wrought and powder specimens ranges from approximately 5 percent at room temperature to 20 to 30 percent at -423°F. More conclusive tests would have to be performed before a conclusion could be drawn as to the suitability of powder for cryogenic applications.

RECOMMENDATIONS

It would be most desirable to obtain valid plain-strain fracture toughness data on wrought and powder specimens. Since it is impractical to work with the large section sizes required for transverse specimens, it is recommended that additional work be confined to the fabrication and testing of longitudinal specimens, for the two titanium alloys, at the three temperatures investigated in the present work. Such long powder specimens can be made with little difficulty by the hot extrusion of canned powders, and there should be no problems in obtaining comparable wrought stock.

It would also be desirable to study the influence of extrusion temperature and heat treatment on the fracture toughness of powder titanium alloys. The combination of fine grain size and chemically homogeneous structure produced in the powder specimens would seem to be a sound basis for the development of maximum fracture toughness at room and at cryogenic temperatures.

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